

This is a digital copy of a book that was preserved for generations on library shelves before it was carefully scanned by Google as part of a project to make the world's books discoverable online.

It has survived long enough for the copyright to expire and the book to enter the public domain. A public domain book is one that was never subject to copyright or whose legal copyright term has expired. Whether a book is in the public domain may vary country to country. Public domain books are our gateways to the past, representing a wealth of history, culture and knowledge that's often difficult to discover.

Marks, notations and other marginalia present in the original volume will appear in this file - a reminder of this book's long journey from the publisher to a library and finally to you.

Usage guidelines

Google is proud to partner with libraries to digitize public domain materials and make them widely accessible. Public domain books belong to the public and we are merely their custodians. Nevertheless, this work is expensive, so in order to keep providing this resource, we have taken steps to prevent abuse by commercial parties, including placing technical restrictions on automated querying.

We also ask that you:

+ *Make non-commercial use of the files* We designed Google Book Search for use by individuals, and we request that you use these files for personal, non-commercial purposes.

+ *Refrain from automated querying* Do not send automated queries of any sort to Google's system: If you are conducting research on machine translation, optical character recognition or other areas where access to a large amount of text is helpful, please contact us. We encourage the use of public domain materials for these purposes and may be able to help.

+ *Maintain attribution* The Google "watermark" you see on each file is essential for informing people about this project and helping them find additional materials through Google Book Search. Please do not remove it.

+ *Keep it legal* Whatever your use, remember that you are responsible for ensuring that what you are doing is legal. Do not assume that just because we believe a book is in the public domain for users in the United States, that the work is also in the public domain for users in other countries. Whether a book is still in copyright varies from country to country, and we can't offer guidance on whether any specific use of any specific book is allowed. Please do not assume that a book's appearance in Google Book Search means it can be used in any manner anywhere in the world. Copyright infringement liability can be quite severe.

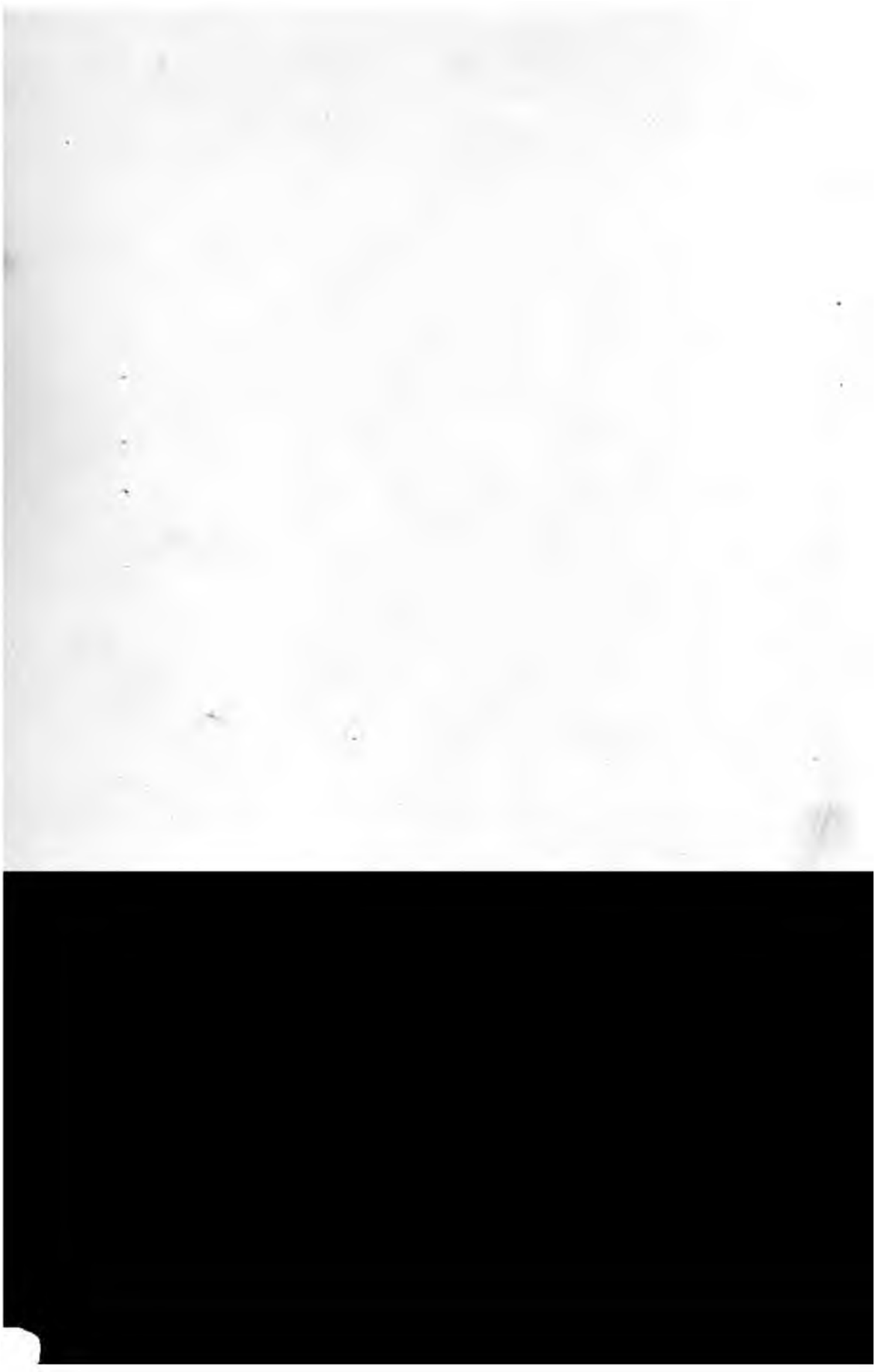
About Google Book Search

Google's mission is to organize the world's information and to make it universally accessible and useful. Google Book Search helps readers discover the world's books while helping authors and publishers reach new audiences. You can search through the full text of this book on the web at `http://books.google.com/`


LANE MEDICAL LIBRARY STANFORD
1601 .L43 1907 STOR
Food inspection and analysis : For the u
24503311845







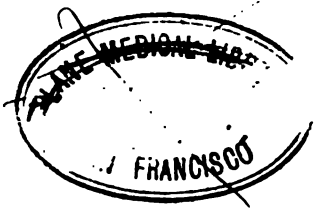
FOOD INSPECTION AND ANALYSIS.

FOR THE USE OF PUBLIC ANALYSTS, HEALTH OFFICERS, SANITARY CHEMISTS, AND FOOD ECONOMISTS.

BY

ALBERT E. LEACH, S.B.,
Analyst of the Massachusetts State Board of Health.





FIRST EDITION.

SECOND THOUSAND.

NEW YORK:
JOHN WILEY & SONS.
LONDON: CHAPMAN & HALL, LIMITED.
1907

Copyright, 1904,
BY
ALBERT E. LEACH.



L43
1907

AFFECTIONATELY DEDICATED TO THE MEMORY OF

Charles Pomeroy Worcester,

FORMER ANALYST OF THE MASSACHUSETTS STATE BOARD OF HEALTH,

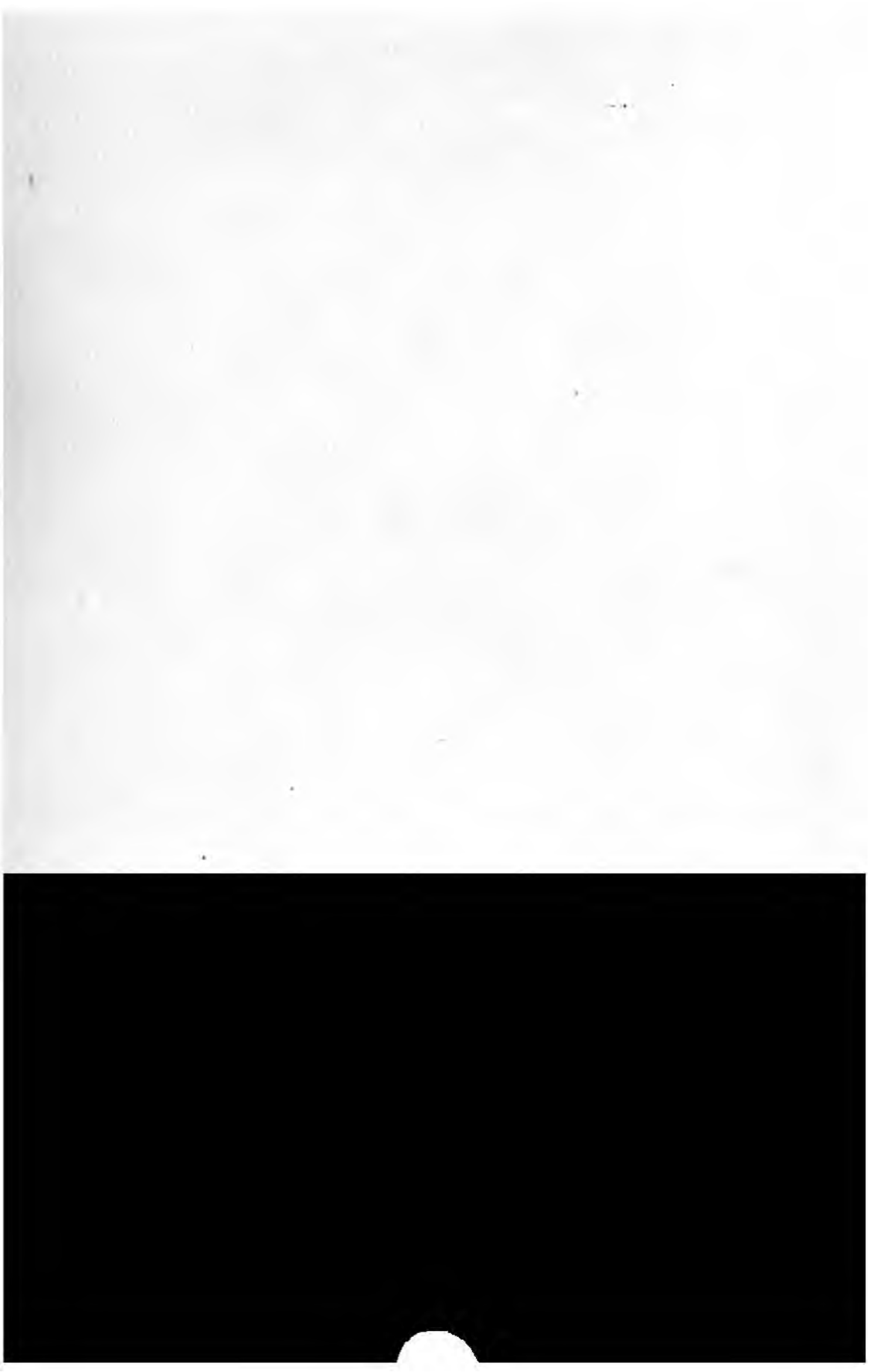
WHOSE LOVABLE PERSONALITY AND STERLING INTEGRITY WERE

A CONSTANT INSPIRATION DURING MANY YEARS

OF CLOSE COMPANIONSHIP TO

THE AUTHOR.

40363



PREFACE.

In the preparation of the present work, the requirements of the public analyst are mainly kept in view, as well as of such officials as naturally cooperate with him in carrying out the provisions of the laws dealing with the suppression of food adulteration in states and municipalities. To this end special prominence is given to the nature and extent of adulteration in the various foods, to methods of analysis for the detection of adulterants, and to some extent also to the machinery of inspection.

While the analyst may not in all cases have directly to deal with the *minutiæ* of food inspection, his work is so closely allied therewith that this branch of the subject is of vital interest and importance to him. Indeed, in many smaller cities one official often has charge of the entire work, combining the duties of both inspector and analyst.

Endeavor has been made, furthermore, to deal with the general composition of foods, and to give such analytical processes as are likely to be needed by the sanitary chemist, or by the student who wishes to determine the proximate components of food materials.

It has been thought best to include brief synopses of processes of manufacture or preparation of certain foods and food materials, in cases where impurities might be suggested incidental to their preparation.

In view of the fact that Massachusetts was the pioneer state to adopt, over twenty years ago, a practical system of food and drug inspection, and for many years was the only state to enjoy such a system, no apology is perhaps needed for more frequent mention of Massachusetts methods and customs than those of many other states, in which the food laws are now being enforced with equal zeal and efficiency.

Considerable attention has been paid in the following pages to the use of the microscope in food analysis. Of the figures in the text illus-

trating the microscopical structure of powdered tea, coffee, cocoa, and the spices, fifteen have been reproduced from the admirable drawings of Dr. Josef Moeller, of the University of Graz, Austria. Acknowledgment is gratefully given Dr. Moeller for his kind consent to their use.

The photomicrographs in half-tone, forming the set of plates at the end of the volume, were all made in the author's laboratory, and may be divided into three classes: 1st, illustrations of powdered pure foods and food products, as well as of powdered adulterants; 2d, types of *adulterated* foods, chosen from samples collected from time to time in the routine course of inspection; and 3d, photographs of permanently mounted *sections* of foods and adulterants.

While recent works covering the whole field of general food analysis are comparatively few, the number of treatises, monographs, government bulletins, and articles scattered through the journals, dealing with special subjects relative to food and its inspection, is surprisingly large, and from a painstaking review of these much information has been culled, for which it has been the author's intention at all times to give credit.

Special mention should here be made of the valuable publications of the U. S. Department of Agriculture, both the bulletins issued from Washington, and those from the various experiment stations, an ever-increasing number of which are becoming engaged in human food work. The author has freely drawn from these sources, and especially from the data and material furnished by his coworkers in the recent and still pending labor of preparing food methods for the Association of Official Agricultural Chemists, and he wishes to extend his thanks to all of them for their assistance. Appreciation is also expressed for the care and discrimination shown by Mr. L. L. Poates in the preparation of the cuts. Thanks are especially due to Mr. Hermann C. Luthgen

TABLE OF CONTENTS.

CHAPTER I.

PAGE

Food Analysis and State Control..... 1–11

Introductory, 1. Food Analysis from the Dietetic Standpoint, 2. Systematic Food Inspection; Functions of the State Analyst; Standards of Purity; Nature of Analytical Methods, 3–5. Adulteration of Food, 5. A Typical System of Food Inspection, 5–8. Practical Enforcement of the Food Laws; Publication; Notification; Prosecution, 9.

References on Food Inspection and State Control, 10.

CHAPTER II.

The Laboratory and its Equipment..... 12–34

Location, 12. Floor; Lighting; Benches, 13. Hoods, 14. Sinks and Drains, 15. Suction and Blast, 17. Apparatus, 18–23. Reagents, 24–30. Equivalents of Standard Solutions; Indicators, 34.

References on Laboratory Equipment, Reagents, etc., 34.

CHAPTER III.

Food, its Functions, Proximate Components, and Nutritive Value..... 35–46

Nature and General Composition of Food, 35. Fats, 35. Protein, and Classification of Nitrogenous Bodies, 36. Proteids, their Subdivisions, Occurrence, and Characteristic Tests, 36–39. Albuminoids, 39. Amido Compounds, 40. Alkaloids; Nitrates; Ammonia; Lecithin, 41. Carbohydrates and their Classification, 41. Organic Acids; Mineral or Inorganic Materials, 42. Fuel Value of Food, 42. The Bomb Calorimeter, 43.

References on Dietetics and Economy of Food, 44.

CHAPTER IV.

General Analytical Methods..... 47–69

Expression of Results, 47. Preparation of Sample, 48. Specific Gravity; Methods, and Apparatus, 49–54. Determination of Moisture; Determination of Ash, 55. Continuous Extraction with Volatile Solvents, 57–60. Separation with Immiscible Solvents, 60. Determination of Nitrogen, 61–63. Determination of Free Ammonia; Determination of Amido Nitrogen, 64. Determination of Carbohydrates, 64. Poisoned Foods, 64. Detection and Determination of Arsenic, 65, 66.

References on General Food Analysis, 67.

CHAPTER V.

PAGE

THE MICROSCOPE IN FOOD ANALYSIS..... 69–87

Microscopical *vs.* Chemical Analysis, 69. Technique of Food Microscopy, 70. Apparatus and Accessories, 70–72. Preparation of Vegetable Foods for Microscopical Examination, 73. Microscopical Diagnosis, 74. Vegetable Tissues and Cell Contents, under the Microscope, 75–78. Microscopical Reagents, 78–81. Microchemical Reactions, 82. Photomicrography; Appurtenances and Methods, 81–86.

References on the Microscope in Food Analysis, 86.

CHAPTER VI.

MILK AND MILK PRODUCTS..... 88–164

Composition and Characteristics of Milk, 88. Milk Sugar; Milk Proteids, and other Nitrogenous Bodies, 89. Milk Fat; Citric Acid; Composition of the Ash, 91. Fore Milk and Strippings, 92. Colostrum; Frozen Milk; Fermentations of Milk, 93. Analysis of Milk, 94. Specific Gravity, 95–97. Total Solids, 97. Ash, 98. Fat, by Extraction, by Centrifugal, and by Refractometric Methods, 98–108. Proteids; Casein, 109. Albumin, 110. Other Nitrogenous Bodies, 111. Milk Sugar, by Optical Methods, 111–113, by Fehling's Solution, 113–115. Relation between the Various Milk Constituents; Calculation by Formulæ, 115–118. Acidity, 117. Boiled Milk, 119. Modified Milk and its Preparation, 119–121. Prepared Milk Foods, Milk Powders, and Artificial Albuminous Foods, 121, 122. Koumis; Kephir, 122.

Milk Adulteration and Inspection; Milk Standards, 123, 124. Forms of Adulteration, and Variation in Standard, 124–126. Rapid Approximate Methods of Examination, 127–129. Systematic Routine Examination, 129. Analytical Methods for Solids, Fat, and Ash, 130–133. Added Foreign Ingredients, 133. Coloring Matters and their Detection, 134–137. Preservatives, their Relative Efficiency and their Detection, 137–145. Added Cane Sugar, 145. Added Starch; Added Condensed Milk, 146. Analysis of Sour Milk, 146.

Condensed Milk; Composition, Standards, Adulteration, 146–148. Methods of Analysis, 148–153. Calculation of Fat in Original Milk, 152.

Cream; Composition, Analytical Methods, Standards, Adulterants, 153–155. Gelatin in Cream, 155; Sucrate of Lime in Cream, 156.

Cheese; Composition, Varieties, 157. Standards; Adulteration, 158. Analytical Methods, 159. Separation and Determination of Nitrogenous Bodies, 160–

PAGE

Meat Extracts; Character and Composition, 192–194. Methods of Analysis, 195. Separation of Nitrogenous Compounds, 195–198.

Fish; Structure, Composition, and Methods of Analysis, 198–199. Crustaceans and Mollusks, 200. Analytical Methods; Preservatives in Fish and Oysters, 201.

Concentrated Foods for Armies and Campers, 201.

References on Flesh Foods, 202.

CHAPTER VIII.

EGGS..... 203–211

Nature and Composition, 203. The Egg White and its Nitrogenous Compounds, 204. Preparation of Albumin, 205. The Egg Yolk and its Composition, 205. Composition of the Ash, 206. Analytical Methods; Determination of Lecithin, 207. Preservation of Eggs, 208. Physical Methods of Examination, 209. Desiccated Egg; Egg Substitutes, 209. Custard Powders, 210.

References on Eggs, 211.

CHAPTER IX.

CEREALS AND THEIR PRODUCTS, LEGUMES, VEGETABLES, AND FRUITS..... 212–275

Composition of Cereals, Vegetables, Fruits, and Nuts, 212–217. Methods of Proximate Analysis, 217–219. Carbohydrates of Cereals, 219. Starch; Detection, Varieties, Classification, Microscopical Examination, 219–223. Starch Determination, by Direct Acid Conversion and by Diastase Methods, 223, 224. Cellulose or Crude Fiber, 224. Pentosans and their Determination, 225, 226. Separation and Determination of the Carbohydrates of Cereals, 227, 228. Proteids of Cereals and Vegetables; Separation and Methods of Analysis, 228–230. Proteids of Wheat, their Separation and Determination, 230, 231. Proteids of Other Cereals and Vegetables, 232, 233. Ash of Cereals and Vegetables, 233, 234.

Flour; Composition; Milling; Inspection, 235. Adulteration, 237. Alum, 237. Gluten Flour, 238. Ergot, 239. Microscopical Examination of Flour and Meal, 239. Wheat Flour, 241. Meal of Rye, Barley, and Oats, 242. Corn Meal, 244.

Bread; Composition; Varieties, 244, 246. Methods of Examination, 246, 247. Adulteration of Bread; Alum, 248. Cake, 249.

Leavening Materials; Yeast, 249. Compressed Yeast; Dry Yeast, 250. Composition and Microscopical Examination, 251. Yeast Testing; Available Carbon Dioxide, 252. Starch in Compressed Yeast, 253.

Chemical Leavening Materials; Baking Powders, their Classification and Composition, 254, 256. Adulteration, 256, 257. Cream of Tartar and its Adulteration, 257. Analysis of Baking Chemicals, 258. Carbon Dioxide, 258–261. Tartaric Acid, 261. Starch, 262. Aluminum Salts, 263. Other Ingredients, 263–265.

Semolina, Maccaroni, and Edible Pastes, 266. Prepared Cereal Breakfast Foods; Nature and Composition, 267, 268. Analytical Methods, 269. Infants' and Invalids' Foods; Classification, 269. Composition, 270. Analytical Methods, 271–273.

References on Cereals, Vegetables, etc., 273.

References on Leavening Materials, 275.

CHAPTER X.

PAGE

TEA, COFFEE, AND COCOA..... 276–309

Tea; Varieties, Method of Manufacture, Composition, 276–279. Analytical Methods, 279. Theine, or Caffeine, 280. Tannin, 281. Extract, 283. Adulteration and Detection of Adulterants, 284. Facing, 284. Spent Leaves; Foreign Leaves, 285. Stems and Fragments, 286. Added Astringents; Tea Tablets, 287. Microscopical Structure, 288.

Coffee; Nature, Composition, Effect of Roasting, 289, 290. Analytical Methods, 291. Adulteration, 292. Microscopical Examination, 293. Chicory; its Microscopical Structure, 294. Composition of Chicory, and its Determination in Coffee, 296. Date Stones, 297. Glazing; Coffee Substitutes, 298.

Cocoa and Cocoa Products; Composition, Methods of Manufacture, 299–301. Theobromine and Nitrogenous Substances, 302. Analytical Methods, 302–304. Cocoa Butter, 304. Adulteration, and Standards of Purity, 304. Addition of Alkali, 305. Microscopical Structure, 306. Cocoa Shells, 307. Added Starch and Sugar, 308.

References on Tea, Coffee, and Cocoa, 308.

CHAPTER XI.

SPICES..... 310–367

Methods of Proximate Analysis Common to all the Spices, 316. Moisture; Ash; Ether, and Alcohol Extract; Nitrogen; Starch; Crude Fiber; Volatile Oils, 311–313. Microscopical Examination, 314.

Cloves; Composition, 314–317. Tannin, 317. Microscopical Examination, 318. Clove Stems, 319. Adulteration and Standard of Purity, 320. Exhausted Cloves, 320. Cocoanut Shells, 321.

Allspice; Composition, 322. Tannin Equivalent, 323. Microscopical Structure, 324. Adulteration and Standard of Purity, 326.

Cassia and Cinnamon; Composition, 326. Microscopical Structure, 328. Adulterants; Standard, 330. Foreign Bark, 330.

Pepper; Composition, 330–333. Nitrogen Determination, 334. Piperin, 335. Microscopical Examination, 335. Adulteration and Standards, 337. Pepper Shells and Dust, 337, 338. Olive Stones, 338. Buckwheat, 339. Long Pepper, 340.

Cayenne; Varieties, Composition, 341. Microscopical Structure, 343. Adulter-



CHAPTER XII.

PAGE

EDIBLE OILS AND FATS. 368–460

Nature and Properties, 368. Fatty Acids, 368, 369. Saponification, 369. Analysis and Judgment as to Purity, 370. Filtering, Weighing, and Measuring Fats, 370. Specific Gravity, 371. Viscosity, 374. Melting-point, 374. Reichert-Meissl Process for Volatile Fatty Acids, 375. Soluble and Insoluble Fatty Acids, 377–379. Saponification Number, 379. Iodine Absorption Number; Hübl's Method, 380. Hanus's Method, 383. Wijs's Method, 384. Bromine Absorption Number, 385. Thermal Tests, 385. Maumené Test; Bromination Test, 386. The Refractometer; Varieties, 389. Butyro-refractometer and its Manipulation, 390–394. Comparative Refractometer Scale, 396. The Abbé Refractometer, 397. Butyro-readings of Fats and Oils, 398. The Acetyl Value, 400. The Valenta and Elaïdin Tests, 402. Free Fatty Acids, 402. Titer Test, 403. Unsaponifiable Matter, 403. Cholesterol and Phytosterol, 404–408. Paraffin, 409. Microscopical Examination of Oils and Fats, 409. Constants of Edible Oils and Fats, 410.

Olive Oil, 412. Composition and Adulteration, 413. Standards, 414. Tests for Adulteration, 415–417. Cottonseed Oil, 417. Bechi's Test, 418. Halphen's Test, 419. Sesame Oil, 419. Adulterants and Tests, 420. Rape Oil, 421. Tests, 422. Corn Oil, 422. Sitosterol, 423. Peanut Oil, 423. Adulterants; Renard's Method, 424. Bellier's Methods, 425. Mustard Oil, 426. Poppyseed Oil, 427. Sunflower Oil, 427. Rosin Oil, 428. Cocoanut Oil, 429. Cocoa Butter; Tallow, 430.

Butter, 430. Composition, 431. Analytical Methods, 432. Ash; Casein; Milk Sugar; Lactic Acid; Salt, 433. Standard Butter Fat, 433. Adulteration, 434. Colors, 434, 435. Preservatives, 436, 437. Renovated or Process Butter; Oleomargarine, 438. Distinguishing Tests for Butter, Process Butter, and Oleomargarine, 440, 441. Butyro-refractometer, 443–445. Reichert-Meissl Number, 445. Specific Gravity; Foam Test; Milk Test, 446. Curd Tests, 447. Microscopical Examination, 448. Foreign Oils; Glucose, 450. Adulteration of Oleomargarine, 451.

Lard, 451. Composition, 452. Lard Oil; Compound Lard, Standards, 453. Adulteration, 453. Foreign Oils, 454. Microscopical Examination, 454. Analysis of Lard and Lard Substitutes, 456.

References on Edible Oils and Fats, 457. References on Butter, 458. References on Lard, 459.

CHAPTER XIII.

SUGAR AND SACCHARINE PRODUCTS. 461–522

Nature and Classification, 461. Cane Sugar, 462. The Sugar Cane; Manufacture of Cane Sugar, 463. Composition of Cane Sugar Products, 464. The Sugar Beet; Manufacture of Beet Sugar, 465. Refining Sugar; Maple Products, 466. Adulteration of Maple Products, 467. Sorghum, 468. Grape Sugar, 468. Levulose; Malt Sugar, 469. Dextrin; Commercial Glucose, 470. Milk Sugar; Raffinose, 472.

The Polariscope and Saccharimetry, 473–478. Comparison of Scales and Normal Weights, 478. Specific Rotary Power; Birotation, 479.

Analysis of Cane Sugar and its Products; Tests for Sucrose, 480. Moisture; Ash; Non-sugars, 481. Sucrose Determination by Polariscope, 481. Inversion, 483. Volumetric Fehling Process, 486. Gravimetric Fehling Methods, 488–497. Electrolytic Apparatus, 493.

PAGE

Analysis of Molasses and Syrups, 498–503. Double Dilution Method of Polarizing, 503. Raffinose Determination, 503. Adulteration of Molasses, and Standards, 504. Glucose Determination, 505. Tin Determination, 507.

Separation and Determination of Various Sugars, 507, 508. Analysis of Glucose, 509. Arsenic in Glucose, 511.

Honey, 511. Composition and Adulteration, 512, 513. Glucose in Honey, 514. Beeswax, 515. Confectionery; Standard; Adulteration, 517. Analysis of Confectionery, 518–521.

References on Sugars, 522.

CHAPTER XIV.

ALCOHOLIC BEVERAGES..... 523–607

Alcoholic Fermentation, 523. Alcoholic Liquors and State Control, 524. Liquor Inspection, 525, 526. Analytical Methods, Common to all Liquors, 527. Detection and Determination of Alcohol, 528. Alcohol Tables, 531–544. The Ebulioscope, 545. Extract; Ash; Artificial Sweeteners, 547.

Fermented Liquors; Cider, 548. Manufacture and Composition, 549–551. Adulteration, 552. Perry, 553. Wine, 554. Classification of Wines, 555. Composition and Varieties, 556–558. Standards, 559. Adulteration, 560–563. Analytical Methods for Wine; Extract; Acidity, 564. Tartaric Acid, 568. Malic Acid, 569. Sugars; Glycerin; Tannin, 570–571. Foreign Colors, 571–573.

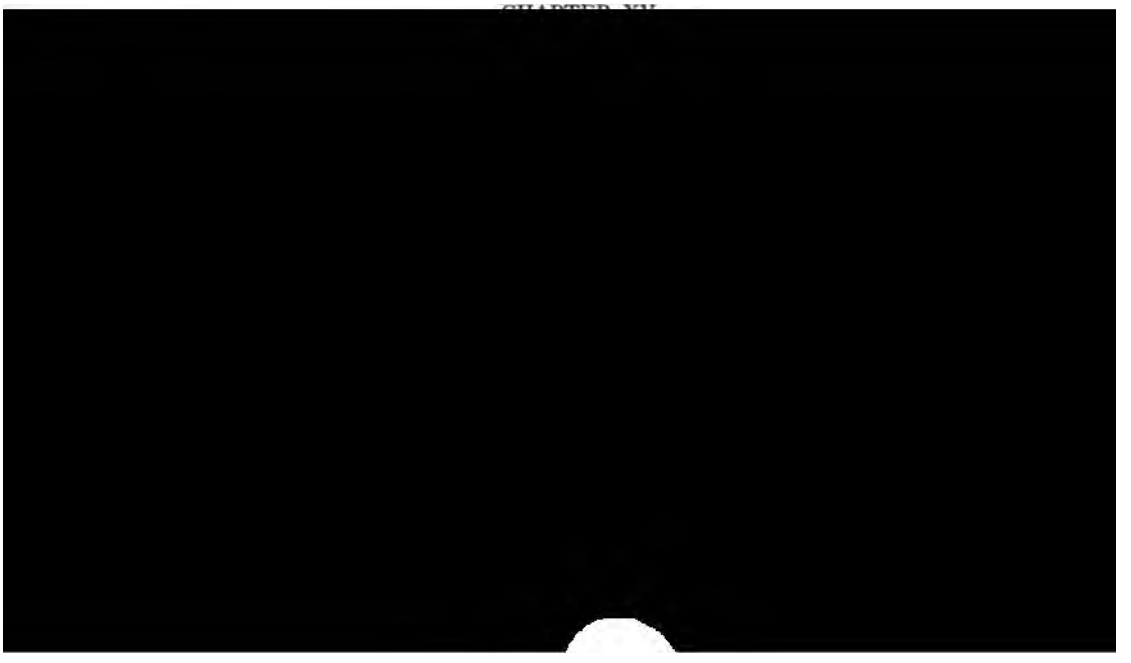
Malt Liquors; Beer, 574. Varieties of Beer and Ale, 575. Composition, 576. Malt and Hop Substitutes, 577. Adulteration and Standards, 578. Malted *vs.* Non-malted Liquors, 578, 579. Preservatives; Arsenic; Temperance Beers, 580. Analytical Methods; Extract; Original Gravity, 581–587. Acids; Proteids; Phosphoric Acid, 588. Carbon Dioxide, 589. Bitter Principles, 590. Arsenic, 591. Malt Extract, 592.

Distilled Liquors, 593. Fusel Oil, 594. Whiskey; Composition, Adulteration, 594–596. Brandy; Manufacture, Adulteration, 597, 598. Rum, 599. Gin, 600. Analytical Methods for Distilled Liquors; Fusel Oil, 601. Ethereal Salts; Furfurol; Caramel, 603.

Liqueurs and Cordials, 605. Analysis of liqueurs, 606.

References on Alcoholic Beverages; on Beer, 607.

References on Cider and Wine; on Distilled Liquors, 608.



CHAPTER XVI.

PAGE

ARTIFICIAL FOOD COLORS..... 628–660

Extent of Use; Objectionable Features, 628. Toxic Effects, 629. Harmful Mineral Colors, 630. Harmful Organic Colors, 631. Harmless Mineral Colors; Harmless Organic Colors, 632–634. Use of Colors in Confectionery, 634. Vegetable Colors, 635. Special Tests; Orchil; Logwood; Turmeric, 637. Caramel; Indigo, 638. Mineral Pigments; Prussian Blue; Ultramarine; Lead Chromate, 638. Cochineal, 638.

Coal-tar Colors, 639. Detection in Food; Basic and Acid Dyes; Wool Dyeing, 640. Double Dyeing Method, 641. Vegetable Colors on Wool, 642. Extraction of Colors by Immiscible Solvents, 642. Separation with Ether, 643. Special Tests, 644. Classification and Identification of Coal-tar Dyes; Rota's Scheme, 644–649. Direct Identification of Colors, 650. Table of Reactions for Colors on the Fiber, 652–659.

References on Colors, 660.

CHAPTER XVII.

FOOD PRESERVATIVES..... 661–681

Preservation of Food, 661. Regulation of Antiseptics, 662. Commercial Food Preservatives, 663. Formaldehyde, 664. Determination in Preservatives, 665. Detection in Food, 666. Determination, 667. Boric Acid; Determination in Preservatives, 667. Detection in Foods, 668. Determination, 669. Salicylic Acid; Detection, 671. Determination, 672. Benzoic Acid; Detection, 673. Sulphurous Acid; Detection, 675. Fluorides, Fluosilicates, Fluoborates, 676, 677. Beta-Naphthol; Detection, 677. Asaprol or Abrastol, 678.

References on Preservatives and their Use in Food, 679.

CHAPTER XVIII.

ARTIFICIAL SWEETENERS..... 682–688

Extent of Use; Saccharin, 682. Detection of Saccharin, 683. Determination, 684. Dulcin; Detection, 685. Determination of Dulcin, 686. Glucin, 687.

References on Artificial Sweeteners, 687.

CHAPTER XIX.

CANNED AND BOTTLED VEGETABLES, RELISHES, AND FRUIT PRODUCTS..... 689–756

Canned Vegetables and Fruits, 689. Method of Canning, 690. Composition, and Methods of Proximate Analysis, 691. Decomposition and Detection of Spoiled Cans, 692. Gases from Spoiled Cans, 693. Metallic Impurities, 694. Action of Fruit Acids on Tin Plate, 695–698. Salts of Lead and Zinc, 699. Salts of Copper; Greening by Copper Salts, 699. Salts of Nickel; Toxic Effects of Metallic Salts, 701. Separation and Determination of Metallic Salts, 701–705. Antiseptics in Canned Foods, 705. Detection of Preservatives, 706. Soaked Goods, 707. Ketchups and Table Sauces, 708. Preservatives in Table Sauces, 710. Pickles and their Adulteration, 711. Horseradish, 712.

Jams and Jellies, 712. Composition and Adulteration, 713–715. Adulterated Jams and Jellies, 716, 717. Labeling "Compound" Goods, 717. Analytical Methods, 718. Determination of Sugars, 719–721. Glucose, 721. Dextrin; Tar-

taric Acid, 722. Citric Acid; Coloring Matters, 723. Preservatives; Starch; Gelatin, 724. Apple Pulp; Microscopical Examination, 725.

Fruit Juices, 725, 726. Sweet Cider; Lime Juice, 727. Fruit Syrups, 728.

Flavoring Extracts, 728. Vanilla Extract, 729. Vanilla Bean; Vanillin, 730. Exhausted Vanilla Bean; Composition of Vanilla Extract, 731. The Tonka Bean; Coumarin, 732. Adulteration of Vanilla Extract; Artificial Extracts, 733. Detection of Artificial Extracts, 734. Methods of Determining Vanillin, 735. Determination of Coumarin, 737. Vanillin and Coumarin under the Microscope; Glycerin, 738. Adulterants, 739.

Lemon Extract, 739–741. Analytical Methods, 741. Determination of Lemon Oil and Alcohol, 742. Methyl Alcohol, 743. Colors; Solids; Ash; Glycerin; Examination of Lemon Oil, 744. Constants of Lemon and Other Essential Oils, 745. Citral, Citronellal, and other Adulterants, 746. Miscellaneous Extracts, 747. Orange Extract, 748.

Almond Extract, 748. Benzaldehyde, 749. Nitrobenzol, 749. Separation and Determination of Benzaldehyde and Nitrobenzol, 750. Alcohol and Hydrocyanic Acid, 751.

Artificial Fruit Essences, 751–753.

References on Canned Foods, Fruit Products, and Flavoring Extracts, 754.

APPENDIX.

The Zeiss Immersion Refractometer, 757–763. Strength of Solutions by the Refractometer, 764. Detection of Added Water in Milk, 765–766. References on Immersion Refractometer, 767.

Detection of Refined Cane Sugar in Maple Products, 767, 768. Polarization of Commercial Glucose, 769. Ash Data of Pure and Adulterated Maple Products, 770.

PLATES I–XL.

PHOTOMICROGRAPHS OF PURE AND ADULTERATED FOODS AND OF ADULTERANTS.

Cereals: Barley, I. Buckwheat, II, III. Corn, III, IV. Oat, IV, V. Rice, V, VI. Rye, VI, VII. Wheat, VIII.

Legumes: Bean, IX. Lentil, IX, X. Pea, X, XI.

Miscellaneous Starches: Potato; Arrowroot; Tapioca, XII. Turmeric; Sago, XIII.



FOOD INSPECTION AND ANALYSIS.

CHAPTER I.

FOOD ANALYSIS AND STATE CONTROL.

INTRODUCTORY.

THE general subject of food analysis, in so far as the public health is concerned, is to be considered from two somewhat different standpoints: first, from the outlook of the government, state, or municipal analyst, whose mission it is to ascertain whether or not the food may properly be considered pure or free from adulteration; and second, from the point of view of the food economist, whose aim is to determine its actual composition and nutritive value. The one protects against fraud and injury, the other furnishes data for the arrangement of dietaries and for an intelligent conception of the rôle which the various nutrients play in the metabolism of matter and energy in the body. The two fields are as a rule distinct each from the other, often involving, in the examination of the food, different methods of procedure.

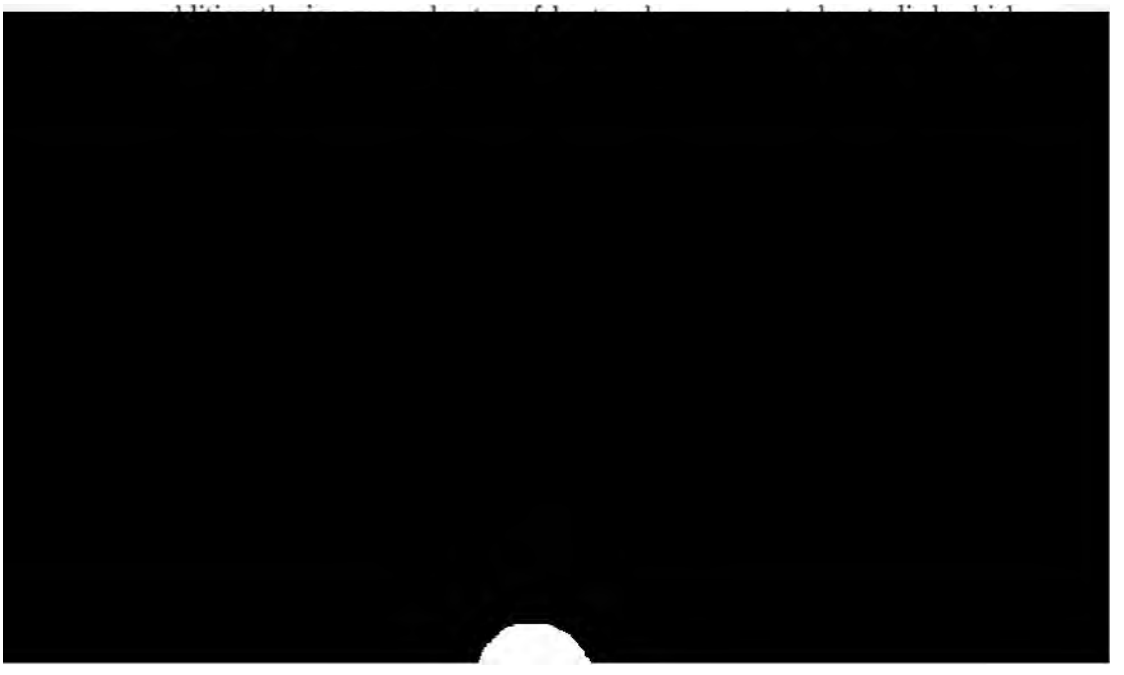
State Control of Food.—In view of the importance of the consideration of food with reference to its purity, an ever-increasing number of states are realizing the necessity of protecting their citizens from the unscrupulous manufacturers who in various lines are seeking to produce cheaper or inferior articles of food in close imitation of pure goods. Many of the states have laws in accordance with which the sale of such impure or adulterated foods is made a criminal offense, and some, but not all of these, are provided with public analysts and other officers to enforce these laws and punish the offenders. Numerous communities are awake to the importance of municipal control of such commonly used articles of food as milk, butter, and vinegar, and in many cases have machinery of their own for regulating the sale of these foods.

Food Analysis from the Dietetic Standpoint.—The study of the principles of dietetics has been given increased attention during the last decade in the curricula of many of the technical schools and colleges. Much has been accomplished by certain of the state experiment stations working as a rule in connection with the United States Department of Agriculture along this line. Investigations of this character are especially valuable, and are indeed rendered necessary by the general tendency of the modern physician to regard the hygienic treatment of disease, especially with reference to the matter of diet, as often of far greater importance than the mere administering of drugs.

The food economist studies the varying conditions of age, sex, occupation, environment, and health among his fellow men, with a view to showing what foods are best adapted to supply the special requirements of various classes. The quantity and proportion of protein, fat and carbohydrates, or of fuel value best suited for the daily consumption of a given class or individual having been determined, dietaries are made up from various food materials to supply the need with reference as far as possible to the taste and means of the consumer.

Experiments are made on families, clubs, or individuals, representing various typical conditions of life, and extending over a given period, during which records are kept of the available food materials on hand and received during the term of the experiment, as well as of those remaining at the end. In the case of individuals, additional records may be kept of the amount and composition of the urine and feces. From such data the physiological chemist calculates the amount of nutrients utilized, and studies the metabolism of material in the human body.

Up to this point no very extensive apparatus is required, but if in



chemical or physical processes involved in making the analyses by which the proximate components of the foods are determined. For more complete information in the field of dietary studies and the metabolism of matter and energy in the body, the student is referred especially to the investigations of Atwater and his coworkers, as published in the annual reports of the Storrs Experiment Station at Middletown, and in the bulletins of the U. S. Department of Agriculture, Office of Experiment Stations, a list of which is given at the end of Chapter III.

SYSTEMATIC FOOD INSPECTION.

The Functions of the State Analyst.—The public analyst is employed by city, state, or government to pass judgment on various articles of food taken from the open market by purchase or seizure, either by himself or by duly authorized collectors employed for the purpose. The sole object of his examination is to ascertain whether or not such articles of food conform to certain standards of purity fixed in some cases by special law, and in others by common usage or acceptance. Such a public analyst need not concern himself with the dietetic value of the food or whether it is of high or low grade. It is for him to determine simply whether it is genuine or adulterated within the meaning of the law, and, if adulterated, how and to what extent. Aside from his skill as a chemist, it is often necessary for him to possess other no less important qualifications, chief among which are his ability to testify clearly and concisely in the courts, and to meet at any time the most rigid kind of cross-examination, it being of the utmost importance that he understand thoroughly the nature of evidence.

U. S. Standards of Purity for Food Products.*—Under an act of Congress approved June 3, 1903, standards of purity for certain articles of food including meat and meat products, milk and its products, sugars and related substances, spices, and cocoa and cocoa products were later (Nov. 20, 1903) established as official standards for the United States by the Secretary of Agriculture. Schedules are yet to be adopted and are in process of preparation in the following classes: grains, fruits and vegetables, honey, fruit extracts, salad oils, salt, tea, coffee, fruit juices, alcoholic and carbonated beverages, vinegar, preservatives, and coloring matters.

Nature of the Analytical Methods Employed.—Usually but a small percentage of the samples submitted for examination are actually adulter-

* U. S. Dept. of Agric., Off. of Sec., Circ. 10.

ated. The analyst should, therefore, adopt for economy in time the quickest possible reliable processes for separating the pure from the impure, so that most of his attention may be devoted to the latter. The nature of the processes by which this is done varies with the foods. Experience soon enables one to judge much by even the characteristics of taste, appearance, and odor, though such superficial indications should be used with discretion. One or two simple chemical or physical tests may often suffice to establish beyond a doubt the purity of the sample, after which no further attention need be paid to it.

A sample failing to conform to the tests of a genuine food must be carefully examined in detail for impurities or adulterants. While in most cases usage or experience suggests the forms of adulteration peculiar to various foods, the analyst should be on the alert to meet new conditions constantly arising. His methods are largely qualitative, since technically he need only show in most cases the mere presence of a forbidden ingredient, though for the analyst's own satisfaction he had best determine the amount, at least approximately.

In reporting approximate quantitative results in court, especially when they are calculated from assumed or variable factors, or when they are the result of judgment based on the appearance of the food under the microscope, the analyst should always be conservative in his figures by expressing the lowest or minimum amount of the adulterant, so as to give the defendant the benefit of any doubt. When exact standards are fixed by law, as in the case of total solids or fat in milk, for example, there is of course great necessity for preciseness in quantitative work.

A full analysis of an adulterated food beyond establishing the nature and amount of the adulteration is entirely unnecessary, and in most instances adds nothing to the strength of a contested case, as twenty

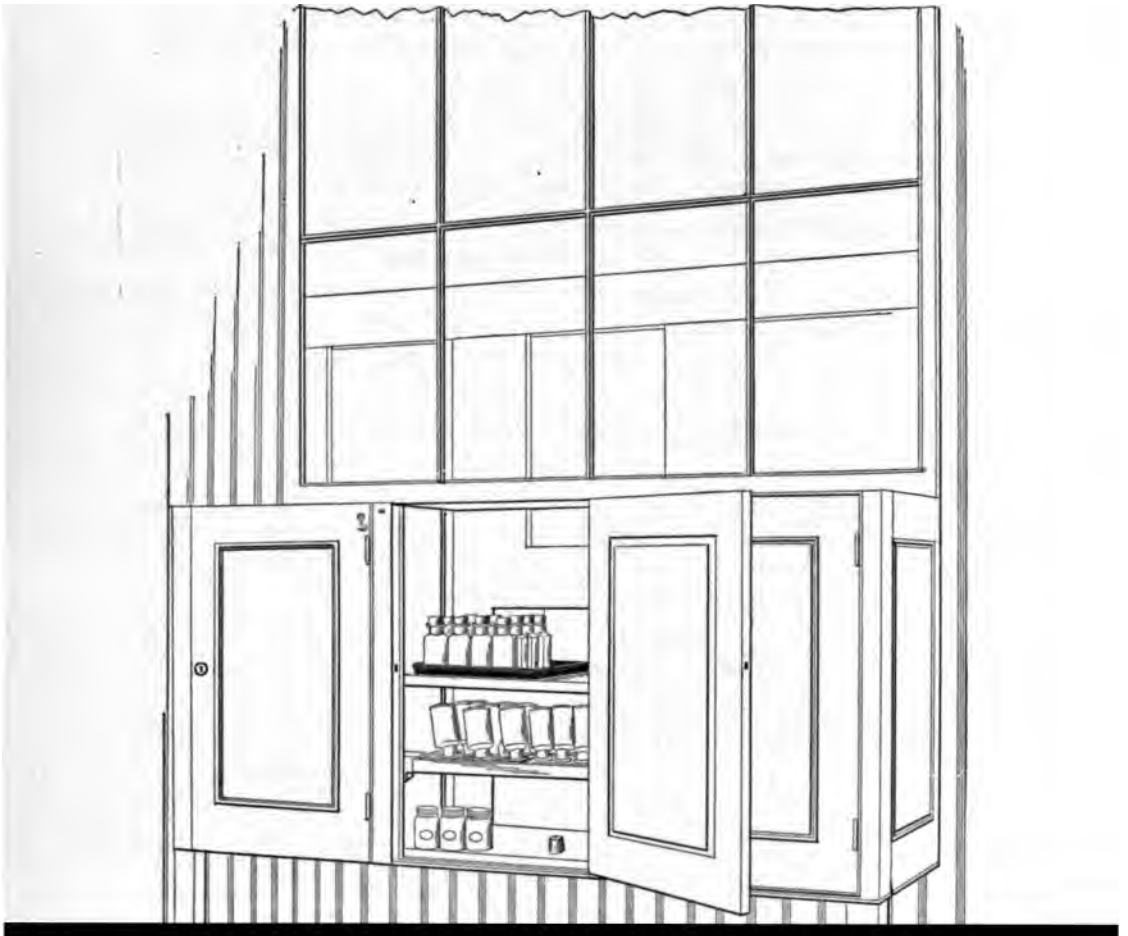
authorities before expressing his opinion; and finally, after being fully convinced that a sample is adulterated, and having so alleged, let him adhere to his statements and not waver in spite of the most rigid cross-examination to which he may be subjected.

While each state or municipality has its own peculiar code of regulations and restrictions concerning the duties of the analyst and other officials, these rules are in the main very similar. For instance, it is usually necessary, excepting in the case of such a perishable food as milk, for the analyst to reserve a portion of a sample before beginning the analysis, which sample, in the event of proving to be adulterated, shall be sealed, so that in case a complaint is made against the vendor, the sealed sample may, on application, be delivered to the defendant or his attorney.

Adulteration of Food.—Except in special cases a food in general is deemed to be adulterated if anything has been mixed with it to reduce or lower its quality or strength; or if anything inferior or cheaper has been substituted wholly or in part therefor; or if any valuable constituent has been abstracted wholly or in part from it; or if it consists wholly or in part of a diseased, decomposed, or putrid animal or vegetable substance; or if by coloring, coating, or otherwise it is made to appear of greater value than it really is; or if it contains any added poisonous ingredient. These provisions briefly expressed are typical of the general food laws found in the statute books of many of the states, though the verbiage may differ. Laws covering compound foods and special foods vary widely with the locality. As to the character of adulteration, nine out of ten adulterated foods are so classed by reason of the addition of cheaper though harmless ingredients added for commercial profit, rather than by the addition of actually poisonous or injurious substances, though occasional instances of the latter are found.

Authentic instances of actual danger to health from the presence of injurious ingredients are extremely rare, so that the question of food adulteration should logically be met largely on the ground of its fraudulent character. Indeed the commoner forms of adulteration are restricted to a comparatively small number of food products, the most staple articles of our food supply, such as flour and the cereals, eggs, fresh meat, fresh vegetables and fruit being rarely subject to adulteration.

A Typical System of Food Inspection.—The efficiency of a system of public food inspection is greatly enhanced if the business part of the work, including the bookkeeping and attending to the outside public, be done wholly through some person other than the analyst, as, for example,



a health officer, to whom the collectors of samples and the analyst may report independently as to the results of their work, and whose duty it is to determine what shall be done in cases of adulteration. In this way

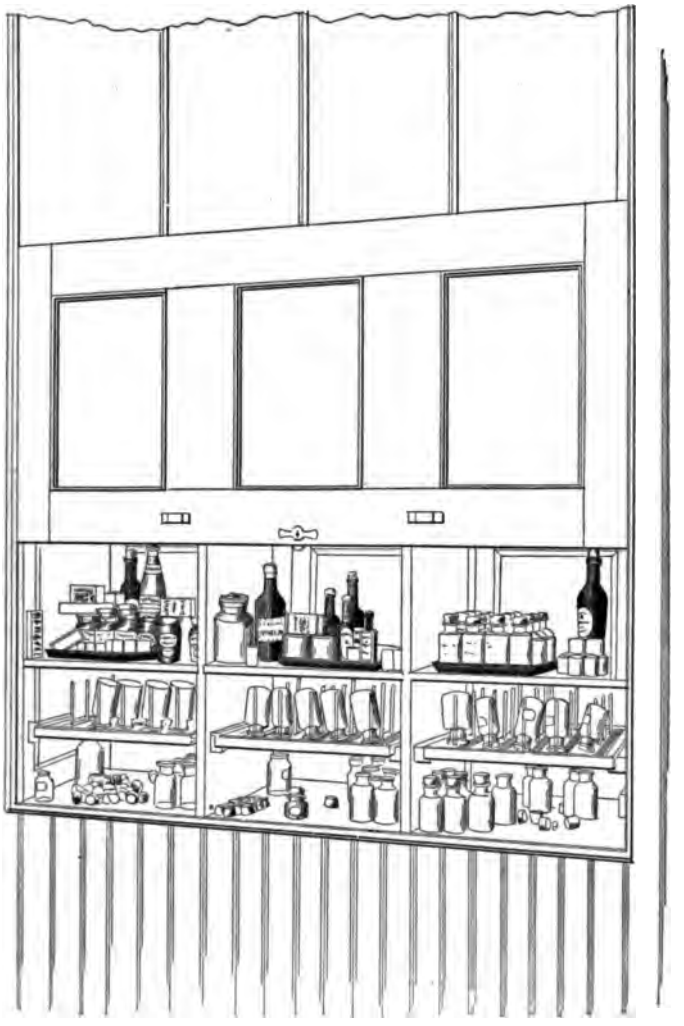


FIG. 2.—Inspectors' Lockers. Front View. The lockers are accessible to the analyst in the laboratory by a single sliding-sash front, provided with a spring lock. The removable sliding-racks are convenient for returning clean sample bottles.

the analyst knows nothing of the data of collection nor the name of the person from whom the sample was purchased, so that he can truthfully state in court that his analysis was unbiased.

Suppose, for example, that three collectors are employed to purchase samples of food for analysis, their duties being to visit at irregular intervals different portions of a state or municipality. Each collector keeps a book in which he enters all data as to the collection of the sample, including the name of the vendor, assigning a number to each sample, which number is the only distinguishing mark for the analyst. One collector may use for this purpose the odd numbers in succession from 1 to 9999, the second the even numbers from 2 to 10,000, while the third may use the numbers from 10,000 up. Each of the two former would begin with a lettered series, as, for instance, A, numbering his samples 1A, 3A, 5A, 7A, etc., or 2A, 4A, 6A, etc., till he reached 10,000, then beginning on series B and so on. If the analyst is to be kept in ignorance of the brand or manufacturer in the case of package goods, the collector must remove from the original package sufficient of the sample for the needs of the analyst, and deliver it to the latter in a plain package, bearing simply the name under which the article was sold and the number. Such precautions are, however, not always practicable and depend largely on local regulations. The analyst reports the result of the analysis of each sample with the number thereof on a library card, with appropriate blanks both for data of analysis and for data of collection, the latter to be filled by the collector from his book after the analyst has handed in the card with the data of analysis. This system of recording and reporting analyses has been successfully used for years by the Department of Food and Drug Inspection of the Massachusetts State Board of Health.

Legal Precautions.—The laboratory of the public analyst should preferably be provided with a locker for each collector, to which access may be had only by that collector and the analyst, so that in the absence of the latter, or when circumstances are such that the samples cannot be



Practical Enforcement of the Food Law.—In the case of foods actually found adulterated, there are three practical methods of suppressing their further sale, viz., by publication, by notification, and by prosecution. These may be separately employed or used in connection with each other, according to the powers conferred by law on the commission, board, or official having in charge the enforcement of the law, and according to the discretion of such official.

Publication.—Under the laws of some states, the only means of protecting the people lies in publishing lists of adulterated foods with their brands and manufacturers' names and addresses in periodical bulletins or reports. Sometimes it is considered best to publish for the information of the public lists of unadulterated brands as well, and, again, it is held that only the offenders should thus be advertised.

Such publication, by keeping the trade informed of the blacklisted brands and manufacturers, certainly has a decidedly beneficial effect in reducing adulteration, and involves less trouble and expense than any other method. It is obviously an advantage, however, in addition to this to be able in certain extreme cases to use more stringent methods when necessary.

Notification and Prosecution.—The adulteration of food is best held in check in localities where under the law cases may be brought in court and are occasionally so brought. The mere power to prosecute is in itself a safeguard, even though that power is not frequently exercised. Under a conservative enforcement of the law, actual prosecution should be made as a last resort. Neither the number of court cases brought by a food commission nor the large ratio of court cases to samples found adulterated are criteria of its good work. Except in extreme cases, it is frequently found far more effective to notify a violator of the law, especially if it is a first offense, giving warning that subsequent infraction will be followed by prosecution. Such a notification frequently serves to stop all further trouble at once and with the minimum of expense. Instances are frequent in Massachusetts where, by such simple notification, widely distributed brands of adulterated foods have been immediately withdrawn from sale.

Massachusetts was the first of all the states to enact pure-food legislation, and for twenty years has enjoyed a well-established system of food inspection, prosecuting cases under its laws through the Food and Drug Department of the State Board of Health. Cases are brought in court with practically no expense for legal services. Complaints are entered by

the collector, or, as he is termed, inspector, who makes complaint not in his official capacity, but as a citizen who under the law has been sold a food found to be adulterated, and who is entitled to conduct his own case, which he does with the aid of the analyst and such other witnesses as he may see fit to employ. Experience is readily acquired by the inspector in conducting such cases in the lower police or municipal courts, where they are first tried, and years ago the services of legal counsel in Massachusetts were dispensed with as superfluous.*

Statistics in the annual reports of the Massachusetts Board show with what uniform success these trials have been conducted. While more often settled in the lower courts, occasional appeal cases are carried to the superior courts, where the services of the regular district attorney are of course availed of in prosecuting the case.

REFERENCES ON FOOD INSPECTION AND STATE CONTROL.

ABBOTT, S. W. Food and Drug Inspection. Article in Reference Handbook of the Medical Sciences, Vol. 3, pp. 162–180. N. Y., 1902.

ANDREWS, O. W. Public Health Laboratory Work and Food Inspection. London, 1901.

BIGELOW, W. D. Foods and Food Control. U. S. Dept. of Agric., Bur. of Chem., Bul. 69.

—— Pure Food Laws of European Countries. U. S. Dept. of Agric., Bur. of Chem., Bul. 61.

BUCHKA, K. VON. Die Nahrungsmittelgesetzgebung im Deutschen Reiche. Berlin, 1901.

CHAPIN, C. V. Municipal Sanitation in the United States. Providence.

KENWOOD, H. R. Public Health Laboratory Work. London, 1904.

LEACH, A. E. Character and Extent of Food and Drug Adulteration in Massachusetts and the System of Inspection of the State Board of Health. Tech. Quarterly, March, 1900.

MOOR, C. D. Suggested Standards of Purity for Foods and Drugs. London, 1902.

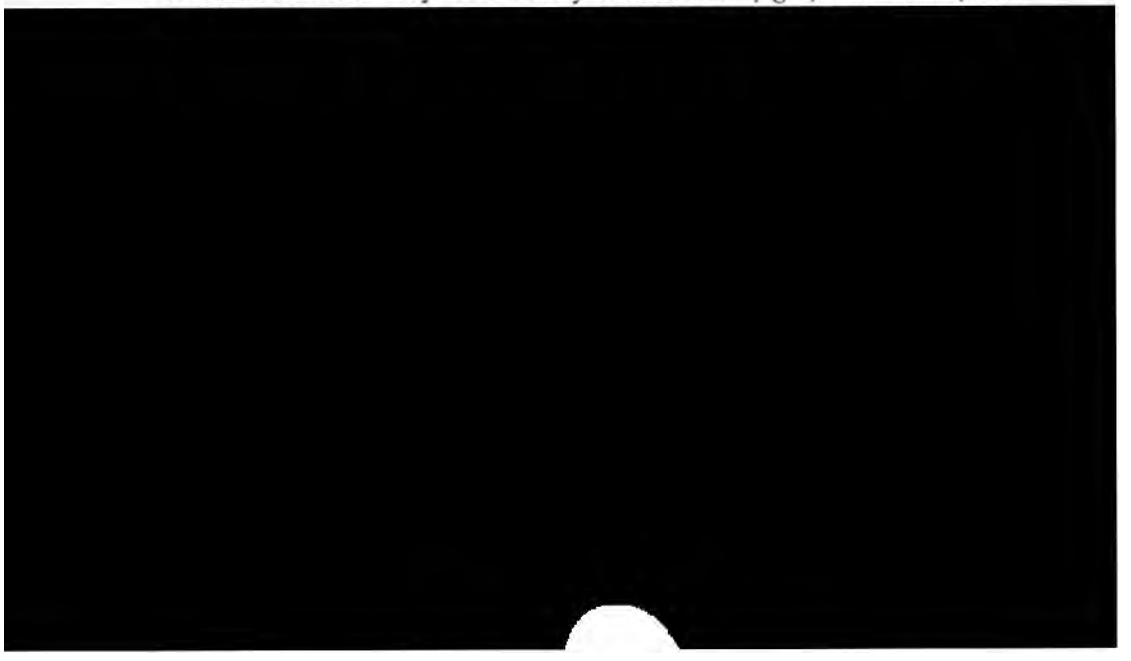


The British Food Journal, London, 1899 et seq.
Food and Sanitation, London, 1892–1900 (discontinued August, 1900).
Journal of the Sanitary Institute, 1892 et seq.
Revue International des Falsifications, Amsterdam, 1888 et seq.
Veröffentlichungen des kaiserlichen Gesundheitsamptes. Berlin, 1877 et seq.
Arbeiten aus dem kaiserlichen Gesundheitsamptes. Berlin, 1886 et seq.
Reports of the Local Government Board of England, 1877 et seq.
Reports of the Paris Municipal Laboratory, 1882 and 1885.
Reports of the Canton Chemists of Switzerland, 1890 et seq.
Annual Reports of the Massachusetts State Board of Health, 1883 et seq.
Monthly Bulletins of the Massachusetts State Board of Health.
Annual Reports of the Conn. Agric. Exp. Station on Food Products, 1896 et seq.
Annual Reports of the Ohio Dairy and Food Commissioner, 1890 et seq.
Annual Reports of the New Jersey Dairy Commissioner, 1886 et seq.
Annual Reports of New Jersey Laboratory of Hygiene, Chemical Dept., 1903 et seq.
Annual Reports of the Michigan Dairy and Food Department, 1893 et seq.
Monthly Bulletins of the Michigan Dairy and Food Department, Aug., 1895 et seq.
Biennial Reports of the Minnesota Dairy and Food Commissioner.
Annual Reports of the Wisconsin Dairy and Food Commissioner, 1890 et seq.
Annual Reports of the Penn. Board of Agriculture, 1894 et seq.
Annual Reports of the Illinois State Food Commissioner, 1899 et seq.
Biennial Reports of the New Hampshire State Board of Health, 1902 et seq.
Quarterly Bulletins of the New Hampshire State Board of Health, 1902 et seq.
Annual Reports of the North Carolina State Board of Agriculture on Food Products, 1900 et seq.
Bulletins of the North Dakota Experiment Station.
Proceedings of the National Association of State Dairy and Food Departments, 1902 et seq.

CHAPTER II.

THE LABORATORY AND ITS EQUIPMENT.

Location.—The selection of a location for a food laboratory cannot always be made solely with reference to its needs and its convenience, but it is more often subject to economic conditions beyond the analyst's control. Under very best conditions, such a laboratory should be situated in a building designed from the start exclusively for chemical or biological and chemical work. Almost any well-lighted rooms in such a building can be readily adapted for the purpose. When, however, as is frequently the case, rooms for such a laboratory are provided in municipal, government, or office buildings, in which for the most part clerical work is done, the problem of adequately utilizing such rooms so that they may not at the same time prove offensive to or interfere with the comfort of other occupants of the building is sometimes difficult. It is obvious that basement rooms in such a building, as far as ventilation is concerned, are less readily adapted for the requirements in hand than are those of the top floor, though, if the light is good and there are abundant and well-arranged ventilating-shafts, such rooms may be made to serve every purpose. In the basement one may most easily obtain water, gas, and steam, and



modified by the means at his disposal, a few general suggestions regarding important essentials may prove helpful: These imply a fairly liberal though not extravagant outlay, with a view to saving both time and energy by convenient surroundings well adapted to the work in hand. At the same time equally satisfactory work is possible under simpler conditions than those described.

Floor.—The best material for the floor of the working laboratory is asphalt. Such a floor is firm but elastic, is readily washed by direct application of running water, if necessary, and resists well the action of ordinary reagents. An occasional thin coating of shellac with lampblack applied with a brush gives the asphalt floor a smooth, hard surface and may be applied locally to cover spots and blemishes.

Lighting.—The lighting of the rooms, if on the top floor, is best effected by both wall windows and skylights. North windows furnish the best light for the microscope; the skylight, when available, is the ideal light for the balance and for general laboratory work.

Ventilation by forced draft is a great convenience. For this purpose an exhaust-fan driven by an electric motor and controlled in speed by a fractional rheostat is admirable. Such a fan had best be located in a small closed compartment or closet near the centre of the series of rooms designed to be ventilated by it, and this closet should have directly over the fan an outlet-shaft passing through the roof of the building. With such a system, a series of branching air-ducts should radiate from the fan closet, conveniently arranged either above or along the ceiling and communicating with the various hoods, closets, and rooms near the top.

Benches.—The working benches should have wooden or glazed tile tops. White glazed tile, if properly laid, furnish a very clean, sanitary, and resistant surface, besides being often convenient for color tests. If laid on a plank surface, the tiles should not be imbedded in cement, which swells the wood before drying out and results in a loose and often uneven surface. For best results the tiles should be first soaked in oil and then carefully laid in putty.

When wooden bench tops are used they may be treated to advantage by staining with the following solutions:

Solution 1.

100 grams of aniline hydrochloride.
40 " " ammonium chloride.
650 " " water.

Solution 2.

100 grams of copper sulphate.
50 " " potassium chlorate.
615 " " water.

The solution should be applied to the bare wood. Soft wood gives a better finish than hard wood. Apply solution 1 thoroughly and allow it to dry; then apply 2 and dry. Repeat these applications several times. Wash with plenty of hot soap solution, let dry and rub well with vaseline.

It is claimed that wood so treated is rendered fire-proof and is not acted on by acids and alkalies. When the finish begins to wear, an application of hot soap solution or vaseline will bring it back to a deep black color.

The benches should naturally be located with reference to best light from skylights or windows. Gas and water outlets, sinks and waste-pipes should be conveniently arranged with reference to the working benches, as well as suitable provisions for air-blast and exhaust, while in the space beneath the benches such drawers, cupboards, and receptacles as are required should be provided. If the benches are excessively wide, there is constant temptation to allow countless pieces of apparatus and reagent bottles not needed for immediate use to accumulate along the back spaces to the detriment of order and cleanliness. A clear bench width of 24 inches is ample for most work. At the back of the bench and within easy reach a raised narrow shelf should be provided to be used *exclusively* for common desk reagents. This again should not be so wide as to allow the accumulation of useless bottles. A narrow raised guard or beading at the edge of the reagent shelf prevents the bottles from accidentally slipping off.



Sinks and Drains.—The sinks should preferably be of iron or porcelain. If iron, they should at frequent intervals be treated with a coat of

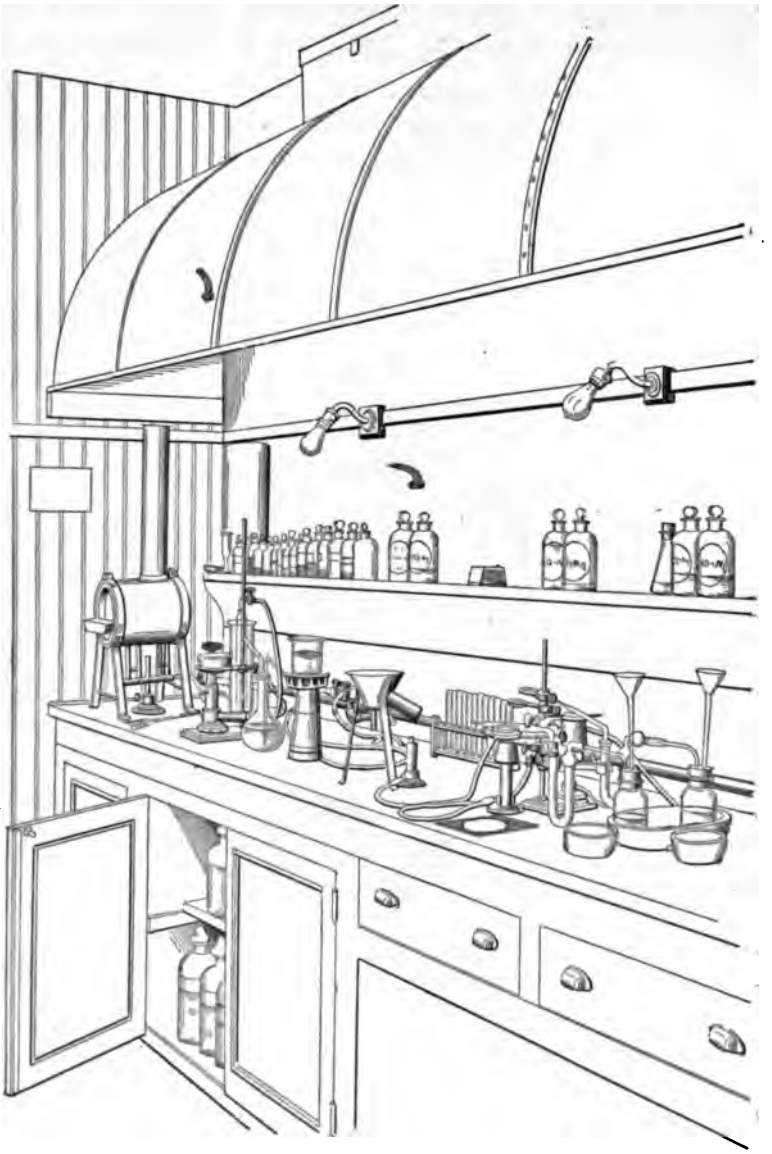
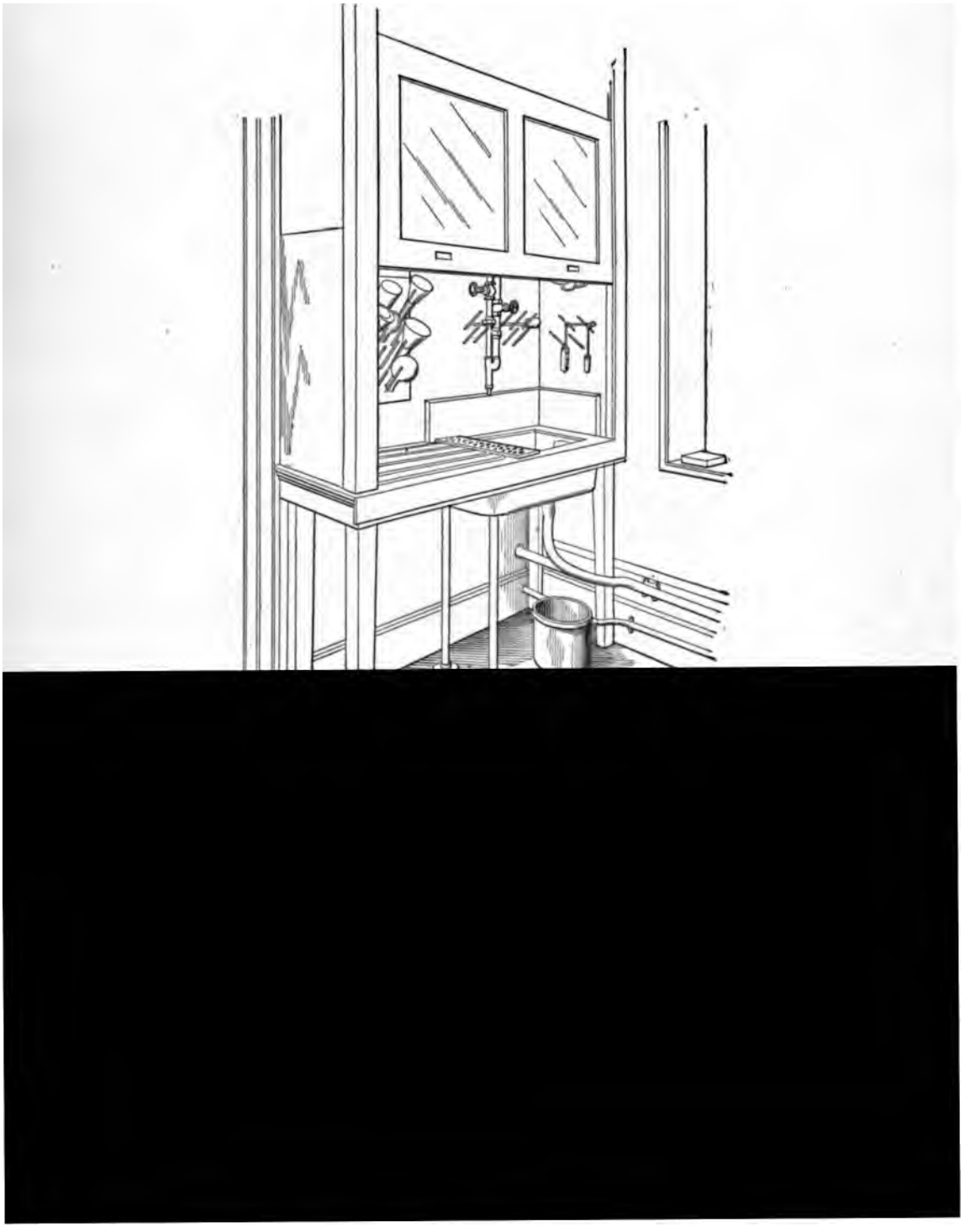


FIG. 3.—Hooded Top of Galvanized Iron over Working-bench, Connected with Ventilating Air-ducts.

asphalt varnish. A great convenience is a hooded sink (Fig. 4) in which foul-smelling bottles, or vessels giving off noxious or offensive fumes

or gases, may be rinsed under the tap while completely closed in. Open-work rubber mats at the bottom of the sinks help to insure against breakage. Open plumbing of simplest design should be used, and a multiplicity of traps should be avoided. Sinks may be variously located for



flush with the top of the floor. Waste-pipes from sinks, still-condensers, refrigerators, and various forms of apparatus involving flowing water may be led into this drain, holes being drilled in the iron cover for their insertion.

Steam and Electricity.—These are useful but not indispensable. Steam, when available, may be used to advantage for boiling ether or benzine in connection with continuous fat-extraction apparatus, for furnishing the motive power for driving the Babcock centrifuge, for heating water-baths and hot closets, and, in connection with cold water, to furnish a

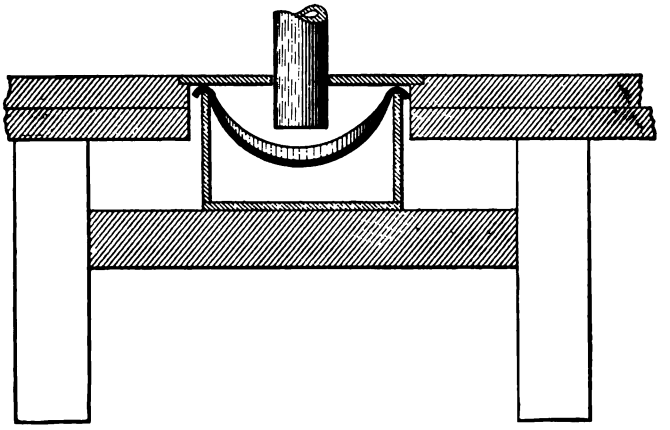


FIG. 5.—Section of Open Drain-pipe in Floor.

supply of hot water when wanted at the sink. The latter application is illustrated in Fig. 4.

If electricity is used for lighting, it may also be applied in a variety of useful ways in the laboratory, as, for instance, for heating coils or electric stoves, for electrolysis, and for running small motors, which in turn may be employed for driving centrifuges, shaking apparatus, ventilating-fans, air-pumps, etc.

Suction and Blast.—If the water-pressure is ample, both air-pressure and exhaust for blast-lamps, vacuum filtration, and other purposes are readily available through the agency of the various devices used in connection with the flow of water, as, for instance, the Richards pump. When, however, the water pressure is insufficient, other means must be employed for furnishing these much-needed requisites. Fig. 6 illustrates a simple and almost noiseless pressure and exhaust pump run by a $\frac{1}{8}$-H.P. electric motor, which with the pressure-equalizing tank and the appropriate connections are mounted on a light wheel truck, and readily movable to any part of the laboratory. By simply screwing the plug into an

electric-light outlet, either suction or blast may be had at will, depending on the position of a knife-edge switch which determines the direction of the current. By means of a fractional rheostat the speed may be varied and the pressure thus controlled.

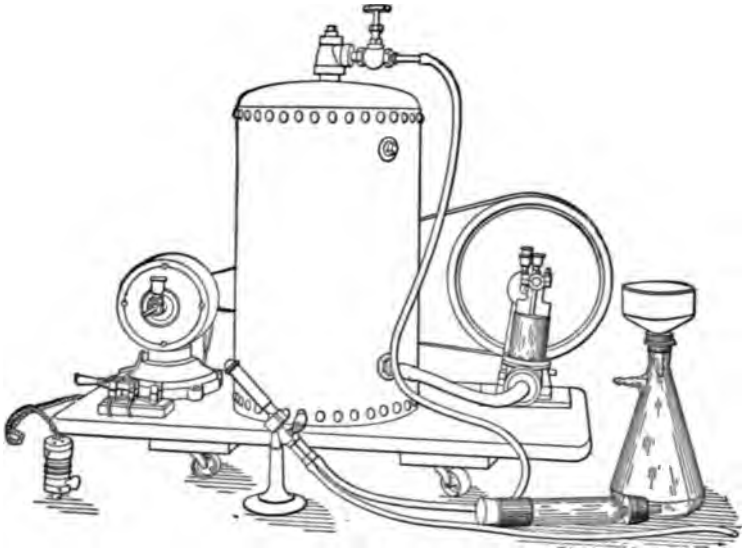


FIG. 6.—Portable Pressure- and Exhaust-pump Run by Electric Motor. Useful for blast-lamps, vacuum filtration, etc.

APPARATUS.

The laboratory is of course to be supplied with the usual assortment of test-tubes, flasks, beakers, evaporating and other dishes of porcelain, platinum and glass, funnels, casseroles, crucibles, mortars, burettes,

able for quick work, and as constructed by the best modern makers leaves nothing to be desired.

The Water-bath.—This is such an important accessory to the food analyst that it should, if possible, be specially designed to meet his require-

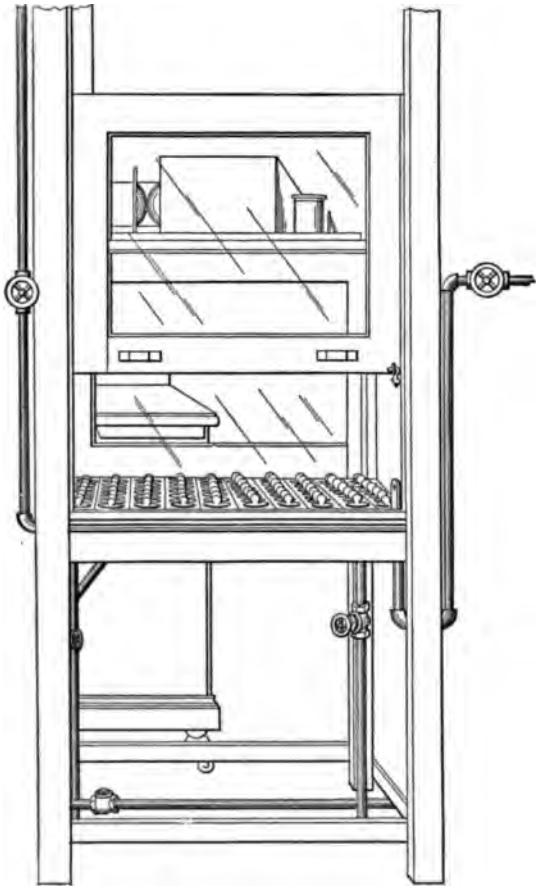


FIG. 7.—Water-bath, Enclosed in Hood, with Sliding-sash Front.

ments, though the ordinary copper baths, supported on legs and designed to be heated by gas-burners, as kept in regular stock by the dealers, will sometimes serve the purpose. For nearly all moisture determinations the platinum dishes described on page 97 and the somewhat larger wine-shells of 100 cc. capacity are most used, and for this purpose the top of the bath should have plenty of openings of the right size for these. A very economical construction of bath admirably adapted for the food analyst's use is shown in plan in Fig. 8, being the form employed by the writer.

The size and number of openings are determined by the number of samples to be simultaneously analyzed. The dotted lines indicate a steam-coil within the body of the bath, which serves to boil the water. Fig. 7 shows the bath in place within a hood, the sliding front of which is furnished with a hasp and padlock, so that it may always be kept locked by the analyst whenever he is temporarily absent from the laboratory. This is a useful precaution, when the residues left thereon are from samples which are to form subjects for possible prosecution in court later.

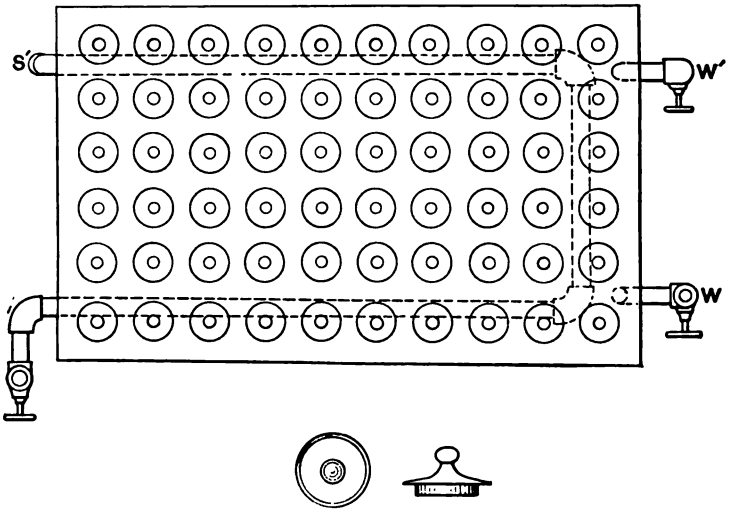


FIG. 8.—Plan View of Water-bath Boiled by Steam-coil. *W* and *W'* are water inlet and outlet, *S* and *S'* steam inlet and outlet, respectively.

Steam, if available at all seasons of the year, furnishes a ready means of heating the bath. Electric immersion coils are also convenient. In the absence of both steam and electricity, the bath must be boiled by gas-

water first serves to cool the condenser, and, rising, becomes vaporized in the boiler directly connected with the condenser at the top. This apparatus is capable of distilling six gallons of water in twelve hours.

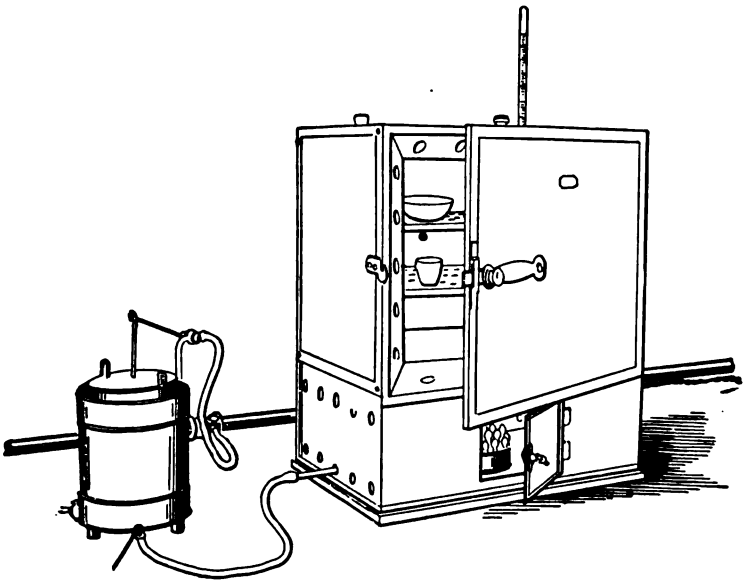


FIG. 9.—Asbestos-covered Air-oven, with Gas-pressure Regulator.

The list of indispensable requisites in addition to the above should include the following:

Continuous Extraction Apparatus (p. 57).
Apparatus for Nitrogen Determination (p. 62).
Apparatus for Distilling Various Food Products (pp. 63 and 530).
A Babcock or other Milk-fat Centrifuge (p. 101).
A Butyro Refractometer (Fig. 91).
A Microscope and its Appurtenances (p. 71).
A Polariscope and its Accessories (Figs. 99, 100, or 101).
Apparatus for Specific Gravity Determination (pp. 50 to 54).
Apparatus for the Determination of Carbon Dioxide (Fig. 65).
Apparatus for the Determination of Melting-points (Fig. 86).
Marsh Arsenic Apparatus (p. 66).
Electrolytic Apparatus (Figs. 108 and 109).
Separatory Funnels (p. 60).

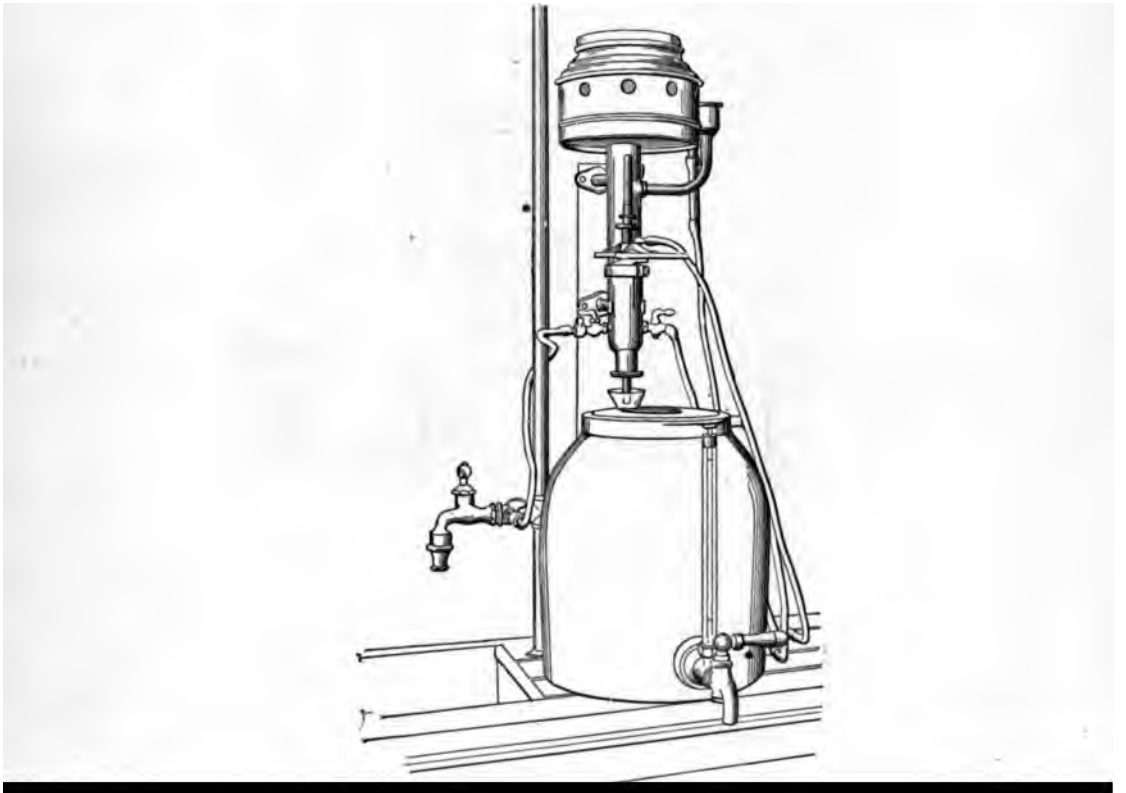
Following is a list of apparatus and appliances which, while not indispensable, are convenient and at times desirable:

A Spectroscope, either of the direct-vision variety for the pocket, or the Kirschoff & Bunsen style on a stand.

Spectroscope Cells, parallel-sided, for observation of absorption spectra.

*A Photomicrographic Camera and Appurtenances** (pp. 84 to 86).

A Muffle Furnace.



A Universal Centrifuge.—This convenient apparatus merits a separate brief description, being useful for a wide variety of purposes, such as breaking up ether- and other emulsions, quickly settling out precipitates, and roughly estimating chlorides, sulphates, phosphates, etc., by the volume of the precipitate in graduated tubes. Various-sized aluminum frames, carrying hinged shields, are interchangeably adjustable to the

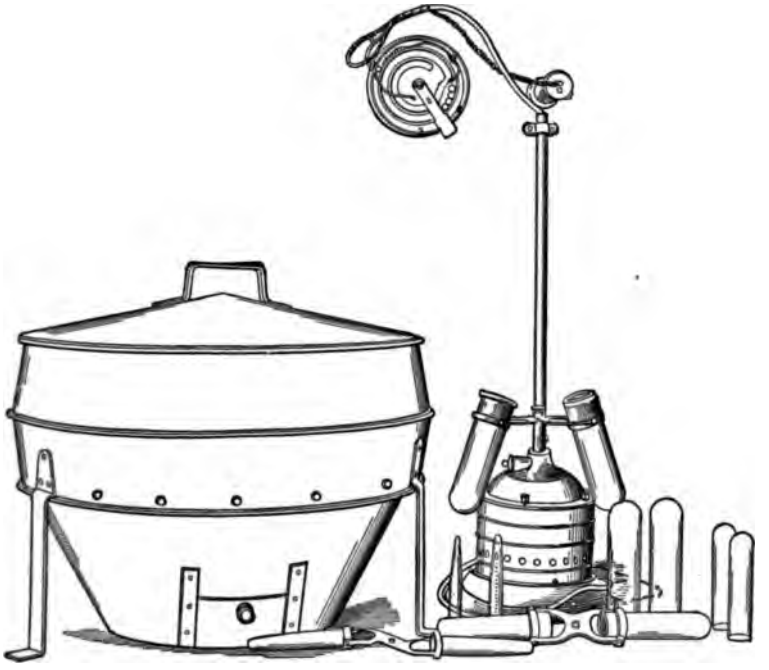


FIG. 11.—The Universal Centrifuge. Driven by an electric motor.

spindle of a vertical electric motor.* The smallest frame has shields adapted to hold two graduated glass tubes of 15 cc. capacity (see Fig. 11). This is for the quantitative estimation of small precipitates and the quick settling of sediments. A medium-sized and large frame carry tubes of 80 cc. and 120 cc. capacity respectively. A frame is also provided with shields adapted for various-sized beakers to be used in settling precipitates. The milk-fat centrifuge frame shown in Fig. 36 is also adapted to be used on the spindle of the same motor.

* In the absence of electricity a water-motor may be used.

REAGENTS.

ula.	Designation.	Molecular Weight.	Grams per Liter.	Remarks, Preparation, etc.
.....	.....	102	.....	Sp. gr. 1.080.
s)	.....	88	.....	" " 0.893 to 0.895. B.P. about 76° C.
.....	Glacial. 99.5%	60	.....	" " 1.055.
.....	30%	.....	.....	" " 1.041.
.....	Crystallized	62		
.....	"	94		
.....	10% solution	.....	100	
.....	C. P. concentrated	36.4	467	Sp. gr. 1.20. 38.92% HCl.
.....	Dilute	.....	265	" " 1.12. 3 vols. No. 8 to 2 vols. H_2O. 23.63% HCl.
.....	10% solution	.....	.....	" " 1.05. 1 vol. No. 8 to 8 vols. H_2O.
.....	N/10 solution	.....	3.64	Standardize against No. 241.
.....	Commercial, conc. ...	.....	.....	Sp. gr. 1.20.
.....	C. P. concentrated ...	63	983	" " 1.42. 69.2% HNO_3.
.....	Dilute	.....	386.5	" " 1.20. 2 vols. No. 13 to 3 vols. H_2O. 32.21% HNO_3.
.....	Commercial conc	.....	.....	" " 1.42.
$+2H_2O$ ...	Crystallized C. P.	126		
.....	N/10 solution	.....	6.3	Weigh freshly crystallized No. 16.
.....	U. S. P. sirupy.....	.....	.....	Sp. gr. 1.725.
H_2O	Crystallized.....	2630		
.....	Solution	.....	50 to 100	Dissolved in 2.5% HCl. See also No. 197.
H.	Crystallized	229		
.....	Solution	.....	.....	Saturated.
.....	Crystallized	126		
.....	"	304		
.....	10% solution.....	.....	100	
H.	Crystallized	138		
.....	C. P. concentrated ...	98	1843	Sp. gr. 1.8426. 100% H_2SO_4.
.....	Dilute	.....	299	" " 1.185. 25.27% H_2SO_4. 1 vol. No. 27 to 5 vols. H_2O.
.....	10%	.....	100	For Reichert number.
.....	1.25%.....	.....	12.5	For crude fiber.
.....	N/10.....	.....	4.9	Standardize against No. 241.
.....	Commercial conc.....	.....	.....	Sp. gr. 1.83 to 1.85 for Babcock process.
.....	Dry.....	322		
.....	.....	88	.....	Sp. gr. 0.818. B.P. 132° C.
.....	Absolute.....	46	.....	" " 0.7938. B.P. 78.4° C.
.....	94% by vol. U. S. P...	.....	.....	" " 0.8200.
.....	70% " "	.....	.....	" " 0.8898. 740 cc. No. 36 per liter.
.....	50% " "	.....	.....	" " 0.9346. 530 cc. No. 36 "
.....	25% " "	.....	.....	" " 0.9711. 266 cc. No. 36 "
.....	10% " "	.....	.....	" " 0.9368. 105 cc. No. 36 "
.....	.....	32	.....	" " 0.814.
.....	Dry root.			
.....	.....	.....	700	In alcohol, see page 80.
$H_4)_2SO_4 + 24H_2O$	Crystallized.....	904.4		

45	Alum (potash)	$Al_2(SO_4)_3.K_2SO_4+24H_2O$	Crystallized	948		
46	"Alumina cream"					Sugar clarifier, page 482.
—	Aluminum hydrate					Same as No. 46.
47	Ammonia	NH_4OH	Concentrated 28%			Sp. gr. 0.901.
48	"		Dilute 10% NH_3			" " 0.9597. 1 vol. No. 47 to 2 vols. H_2O.
49	Ammonium carbonate	$(NH_4)_2CO_3.H_2O$	Crystallized	114		
50	" "		Solution		250	
51	" chloride	NH_4Cl	Dry salt	53.38		
52	" "		Solution		100	
53	" molybdate	$(NH_4)_2MoO_4$	"			100 grams MoO_3 No. 185 mixed with 400 cc. cold H_2O and 80 cc. ammonia No. 47. Filter and add filtrate to 300 cc. HNO_3 No. 13 and 700 cc. H_2O.
54	" nitrate	NH_4NO_3	Dry salt	80		
55	" oxalate	$(NH_4)_2C_2O_4.H_2O$	Crystallized	142		
56	" "		Solution		40	
57	" phosphate	$(NH_4)_2HPO_4$	Crystallized	132		
58	" sulphate	$(NH_4)_2SO_4$	"	132		
59	" "		Solution			Saturated.
60	" sulphide	$(NH_4)_2S$	"			Saturate 3 vols. No. 48 with H_2S and add 2 vols. No. 48.
61	" sulphocyanate	NH_4CNS		76		
62	" "		N/10			See page 141.
63	Animal charcoal		Dry			Pulverized bone charcoal, page 502.
64	Aniline	$C_6H_5.NH_2$	Liquid	93		
65	Asbestos fiber		Finely disintegrated			Washed in acid and alkali, page 489.
66	Arsenious oxide	As_2O_3	Dry C. P. substance	198		
67	" "		N/10		4.95	Dissolve 4.95 grams As_2O_3 No. 66 and 20 grams K_2CO_3 in 250 cc. H_2O. Dilute to 1 liter.
68	Badouin's reagent					1 gram sucrose in 10 cc. HCl No. 8.
69	Barfoed's copper acetete reag't					14 grams crystallized copper acetate No. 104 and 5 cc. acetic acid No. 4 in 200 cc. H_2O.
70	Barium carbonate	$BaCO_3$	Dry salt	197		
71	" chloride	$BaCl_2.2H_2O$	Crystallized	244		
72	" "		Solution		100	
73	" nitrate	$Ba(NO_3)_2$	Dry salt	261		
74	" "		Solution		75	
75	Bechi's reagent					2 grams $AgNO_3$ in 200 cc. alcohol. No. 36, and 40 cc. ether. Add drop HNO_3.
—	Benzine					See petroleum ether, Nos. 190–192.
76	Bismuth solution (alkaline)					4 grams bismuth subnitrate, 4 grams Rochelle salt, 8 grams sodium hydroxide dissolved in 100 cc. water by aid of heat.
77	Bismuth subnitrate	$BiO.NO_3+H_2O$	Crystalline powder.			
—	Bleaching powder					See chlorinated lime.
78	Borax	$Na_2B_4O_7.10H_2O$	Crystallized	382		
79	Bromine	Br	Liquid	80		
80	" water					Saturate H_2O with bromine, having a few drops bromine in excess in reagent bottle.
81	Brücke's reagent					See page 188.
82	Calcium chloride	$CaCl_2.6H_2O$	Crystallized	219		

REAGENTS—(*Continued*).

Formula.	Designation.	Molecular Weight.	Grams per Liter.	Remarks, Preparation, etc.
.....	Solution.....	.....	125	
.....	Anhydrous. Granular	.....	.....	For absorbing moisture.
$_2$.....	Slaked lime	74		
.....	.....	.....	.....	See chlorinated lime, No. 95.
.....	Quicklime.....	56		
H_2O.	Dry salt	172		
.....	Solution.	.....	.....	A saturated solution.
.....	.....	.....	.....	A solution in xylol.
.....	.....	.....	.....	See sucrose.
.....	Liquid.....	76		
.....	.....	153.6		
$.H_2O$.....	Crystallized.....	165		
.....	Solution.....	.....	600	
.....	Gas	35.45	.....	(1) Heat MnO_2 with HCl. (2) Treat chlorinated lime with H_2SO_4 No. 28.
.....	.....	.....	.....	Calcium hydrate saturated with Cl.
.....	.....	.....	.....	H_2O saturated with Cl.
.....	.....	119.1		
.....	.....	.....	.....	See page 79.
$)_2.6H_2O$.	Crystals	290.1		
.....	Solution	.....	50	
.....	Dry.			
.....	Solution.	.....	12	Indicator. Macerate in 25% alcohol. Filter, and neutralize the filtrate.
.....	.....	.....	.....	See page 572.
$_2O_2)_2.H_2O$.....	Crystallized	199		
.....	.....	63.5		
$.H_2O$.....	Crystallized.....	249.5		
.....	.....			
$H_2.CO_2$.....	Crystallized.....	146		
.....	.....	.....	.....	See page 81.
.....	.....	18		
$_4O_5$.....	.....	.....	.....	See page 251.
O.....	U. S. P.....	74	.....	Sp. gr. 0.725 to 0.728. About 96% ether and 4% alcohol, with a little water.
.....	Absolute.....	.....	.....	See page 58.
.....	.....	.....	.....	See No. 1.
.....	.....	.....	.....	34.639 grams pure $CuSO_4$ 5 H_2O in 500 cc. H_2O.
.....	.....	.....	.....	178 gms. Rochelle salts and 50 gms. NaOH in 500 cc. H_2O.
$_2O_2)_6$.....	Dry salt	466		
.....	Solution.....	.....	100	
$_2H_2O$.....	.....	541		
.....	Solution.....	.....	100	
$_7H_2O$.....	Crystallized.....	278		

121	Ferrous sulphate		Solution		100	Prepare fresh for use.
122	" sulphide	FeS		88		
123	Formaldehyde		Solution			40% known as formalin.
124	Fuchsin	$C_{20}H_{19}N_3HCl$	Crystallized.			
125	Fuller's earth					See page 603.
126	Furfurol	$C_4H_3O.COH$		96		
127	Gelatin					
128	"		Solution			See page 281.
129	Glycerin	$C_3H_5(OH)_3$	U. S. P.	92		
130	Glycerin jelly					Microscopic mountant, see page 74.
131	Halphen's reagent					Equal vols. amyl alcohol and CS_2 (containing 1% sulphur in solution).
132	Hanus' iodine reagent					See page 383.
133	Hydrochloric acid: ferric chloride reagent					Conc. commercial HCl, with 2.5 cc. No. 119 per liter.
134	Hydrogen dioxide	H_2O_2		34		
135	" sulphide	H_2S	Gas	34		Treat FeS with dilute H_2SO_4.
136	" " water					Saturate H_2O with H_2S.
137	Hübl's iodine reagent					See page 380.
138	Indigo carmine					
139	"		Solution			See page 281.
140	Indol	C_8H_7N	Crystals	234		See page 80.
141	Iodine	I	Resublimed	126.6		
142	"		N/10 solution		12.66	Triturate I in mortar with 18 grams KI and small portions H_2O till all is dissolved. Dilute to 1 liter, standardizing against No. 245.
143	" in potassium iodide		Solution			2 grams crystallized KI dissolved in 100 cc. H_2O. Saturate solution with I.
144	" trichloride	ICl_3		232.7		
145	Iron wire					Of known purity.
146	Kaolin		Powdered.			
147	Lacmoid		Dry powder.			
148	"		Indicator solution		2	In 50% alcohol. For indicator.
149	Lactose	$C_{12}H_{22}O_{11}.H_2O$	Dry powder	360		
150	Lead acetate	$Pb(C_2H_3O_2)_2.3H_2O$	Crystallized	379		
151	" "		Solution		100	
152	" hydrate					See page 735.
153	" subacetate		Use U. S. P. solution			Or prepare as on page 481.
154	Ligroin					See petroleum ether.
155	Lime water					Saturate H_2O with $Ca(OH)_2$.
156	Litmus		Dry color.			
157	"		Tincture			Digest 1 part litmus in 6 parts H_2O. Filter and divide into 2 parts. Slightly acidify 1 part by glass rod dipped in H_2SO_4 (dil.) to faint red. Add other or blue portion of filtrate.
158	Litmus paper					Dip strips of filter-paper in No. 157.
159	Logwood		Dry color.			
160	"		Solution		50	In alcohol.
161	Magnesium carbonate	$MgCO_3$		84		
162	" chloride	$MgCl_2.6H_2O$	Crystallized	203.3		

REAGENTS—(*Continued*).

ula.	Designation.	Molecular Weight.	Grams per Liter.	Remarks, Preparation, etc.
.....	.....	40.3		
.....	.....	.....	.....	Dissolve 100 grams $MgCl_2$ in H_2O. Add NH_4OH in excess, and NH_4Cl till precipitate is redissolved. Dilute to 1 liter.
.....	Crystallized	246.3		
.....	Pulverized	87		
.....	.....	.....	.....	Page 434.
.....				
.....	.....	.....	.....	Pages 185, 224.
.....	.....	.....	.....	13.525 grams $HgCl_2$ and 49.62 grams KI in 1 liter of water.
.....	.....	200		
.....	.....	271		
.....	Saturated solution ...	.....	.....	Saturate H_2O with $HgCl_2$.
.....	Dilute solution.....	.....	50	
.....	Dry salt	216		
.....	" "	296		
.....	Solution, strong.....	.....	.....	See page 111, sugar clarifier.
.....	" dilute.....	.....	.....	See page 156.
.....	.....	.....	.....	90% ethyl alcohol No. 36 and 10% methyl alcohol No. 41.
.....				
.....	Dry color.			
.....	Indicator solution....	.....	.....	In H_2O.
.....	.....	.....	.....	Free from formaldehyde.
.....	.....	.....	.....	See No. 155.
.....	.....	.....	.....	See No. 149.
.....	.....	.....	.....	Dissolve Hg in twice its weight of HNO_3 No. 13 and dilute with equal vol. of H_2O.
.....	Dry substance.....	143.8		
.....	.....	.....	.....	See Nos. 190–192.
.....	Dry color.			
.....	.....	.....	.....	Dissolve 50 grams KI in H_2O, add saturated $HgCl_2$ solution No. 173 to a distinct red precipitate. Add 350 cc. of a solution of KOH containing 500 grams per liter, make up to a liter and allow to settle, using clear solution.
.....	.....	.....	.....	Page 422.
.....				
.....	.....	.....	.....	B.P. 35° C. and under for extraction in separatory funnel.
.....	.....	.....	.....	B.P. 35° to 50° C. for Soxhlet extraction.
.....	.....	.....	.....	B.P. 50° C. and over for cleansing.
.....	.....	.....	.....	See No. 6.
.....	.....	.....	.....	See page 80.
.....	Dry powder.....	318		
.....	Indicator solution....	.....	1	Dissolve in 50% alcohol.

197	Phosphotungstic reagent.					Dissolve 120 grams sodium phosphate and 200 of sodium tungstate in 1 liter of H_2O and add 100 cc. of conc. H_2SO_4. See also No. 20.
198	Platinic chloride.	$H_2PtCl_6.6H_2O$	Crystallized.			Properly hydrochloroplatinic acid.
199	" "		Solution.		50	
200	Potassium bichromate.	$K_2Cr_2O_7$	Dry salt.	295		
201	" "		N/10 solution		4.9	Dissolve in H_2O and dilute to 1 liter.
202	Potassium bisulphate.	$KHSO_4$	Dry salt.	136		
203	" carbonate.	K_2CO_3	" "	138		
204	" chlorate.	$KClO_3$	Crystallized salt	122.5		
205	" chromate.	K_2CrO_4	" "	194.5		
206	" "		Solution		100	
207	" cyanide.	KCN	Dry salt.	65		
208	" "		Solution		13	See page 141.
209	" ferricyanide	$K_3Fe(CN)_6$	Dry salt.	329		
210	" "		Solution		50	Prepare freshly for use.
211	" ferrocyanide	$K_4Fe(CN)_6.3H_2O$	Crystallized.	422		
212	" "		Solution		50	
213	" hydroxide	KOH	Dry substance.	56		
214	" "		Solution.		500	
215	" "		"		1000	For CO_2 determination.
216	" iodide.	KI	Crystallized.	166		
217	" "		Solution.		100	
218	" nitrate.	KNO_3	Crystallized.	101		
219	" permanganate	$KMnO_4$	"	158		
220	" "		N/10 solution.		3.16	Adjust strength of solution by repeated trial, standardizing against No. 145.
221	" phosphate.	K_3PO_4	Crystallized.	211.9		
222	" sulphate	K_2SO_4	"	174		
223	Resorcin.	$C_6H_4(OH)_2$	"	110		
224	"		Solution		100	
225	Rochelle salt.	$KNaC_4H_4O_6.4H_2O$	Crystallized.	282		
226	Schiff's reagent.					Dissolve 1 gram of fuchsin in H_2O, add a mixture of 20 cc. $NaHSO_3$ solution No. 234 and 10 cc. HCl No. 8. Make up to 1 liter.
227	Schultze's reagent.					See page 81.
228	Silver nitrate.	$AgNO_3$	Crystallized.	170		
229	" "		Solution.		25	
230	" "		N/10 solution		17	Standardize against No. 238.
231	Soda lime.					See page 258.
232	Sodium	Na.	Metal	23		Keep under petroleum.
—	" and potassium tartrate					See Rochelle salt, No. 225.
233	" bisulphite.	$NaHSO_3.H_2O$.	Crystallized.	104		
234	" "		Solution.			Saturated.
—	" borate.					See No. 78.
235	" carbonate.	Na_2CO_3.	Dry substance.	106		
236	" chloride.	NaCl.	Crystallized, C. P.	58.5		
237	" "		Solution.		150	
238	" "		N/10 solution.		5.85	
239	" hydroxide	NaOH	Dry substance.	40		

REAGENTS—(*Continued*).

Formula.	Designation.	Molecular Weight.	Grams per Liter.	Remarks, Preparation, etc.
.....	Solution.	.....	500	
.....	N/10 solution	.....	4	Adjust strength of solution by repeated trial, standardizing against No. 17.
.....	Solution	.....	.....	Agitate 1 part chlorinated lime, No. 95, with 10 parts H_2O. Add $NaCO_3$ as long as precipitate forms. Let precipitate subside, and siphon off supernatant liquid.
$O_4.12H_2O$.	Crystallized	358		
$.5H_2O$	Crystallized C. P.....	248	.....	Guaranteed reagent. Free from sulphate.
.....	N/10 solution.....	.....	24.8	
$.H_2O$.	Crystallized.....	311.6		
H_2O.	Solution	.....	.....	Saturate conc. HCl with tin, dilute with an equal vol. of H_2O, and from time to time add slight excess of acid. Keep pieces of tin in reagent bottle.
.....	Indicator solution....	.....	10	Mix into thin paste with cold H_2O. Pour into 100 times its weight of boiling H_2O. Boil, settle, and use supernatant liquid.
ll	Crystallized	.....	.....	Powdered rock candy.
.....	.....	.....	.....	Page 318.
.....	.....	.....	.....	See acid, tannic, No. 33.
.....	Granulated.....	118		
.....	.....	.....	.....	See page 420.
.....	Dry powder,			
.....	.....	.....	.....	Make strong alcoholic solution of No 254, dip strips of filter-paper therein, and dry.
$_2H_3O_2)_2.2H_2O$..	Crystallized	426		
.....	Solution.....	.....	.....	See page 588.
$.OCH_3.CHO$...	Crystallized	152		
.....	Solution.....	.....	.....	See page 735.
.....	.....	.....	.....	See page 421.
.....	.....	.....	.....	See page 384.
.....	.....	.....	.....	As a medium for Canada balsam.
.....	Granulated.....	65.1		
$_3O_2)_2.3H_2O$.	.....	237		
.....	.....	.....	.....	See No. 98.
H_2O.	Crystallized	287		
.....	Solution.....	.....	.....	A saturated aqueous solution of the salt No. 265 acidified with H_2SO_4.

REAGENTS.

The foregoing list includes the general reagents used in carrying out the processes treated of in this volume, together with their strength, mode of preparation when necessary, and other data. Reagents, especially those constantly employed, should be assigned to regular places on the shelves, and should invariably be kept in place when not in use.

Among the standard solutions for volumetric work, none is more frequently of service in the food laboratory than a tenth-normal solution of sodium hydroxide, and a large supply of this reagent, carefully standardized, should be at all times conveniently at hand. Besides being useful for standardizing tenth-normal solutions, it is constantly needed for determining various acids in food products, such as milk, vinegar, butter, lime juice, cream of tartar, liquors, and many others. Time is well spent in carefully adjusting this solution to its exact tenth-normal value, thus simplifying the calculation of results. A large stock bottle (say of two gallons capacity) containing the standard tenth-normal sodium hydroxide, is conveniently mounted with a side-tube burette in connection, in some such manner as shown in Fig. 12. A small connecting side bottle contains a strong solution of sodium hydroxide (reagent No. 240) through which the air that enters the large bottle is passed, thus depriving it of CO_2. In this manner the standard solution may readily be kept of unvarying strength for a year or more.

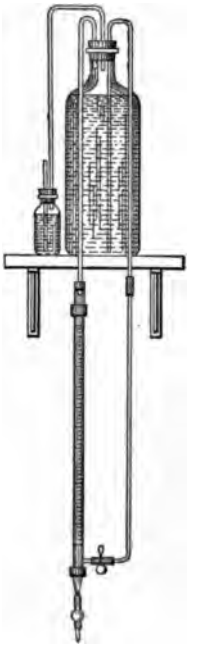


FIG. 12.—Stock Bottle of Tenth-normal Alkali.

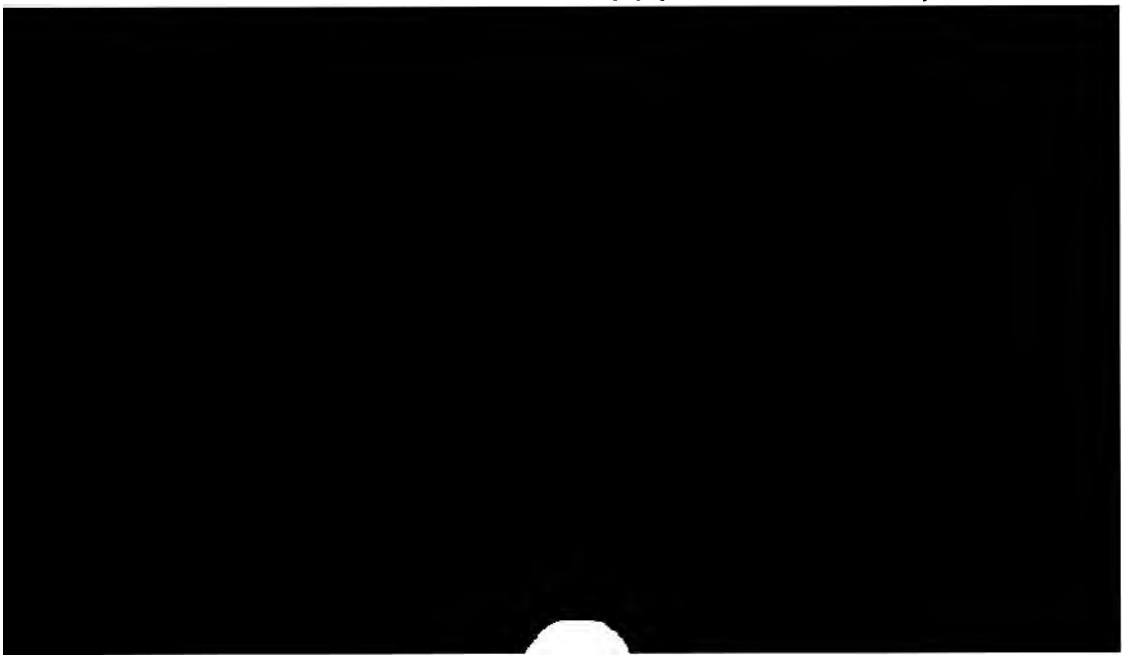
EQUIVALENTS OF STANDARD SOLUTIONS.

No. 31. DECINORMAL SULPHURIC ACID. One cc. is equivalent to

Ammonia gas	NH_3	0.0017 gram.
Ammonia	NH_4OH	0.0035 "
Ammonium carbonate	$(NH_4)_2CO_3$	0.0046 "
" "	$(NH_4)_2CO_3H_2O$	0.0057 "
Calcium carbonate	$CaCO_3$	0.0050 "
Calcium hydroxide	$Ca(OH)_2$	0.0037 "
" oxide	CaO	0.0028 "
Lead acetate	$Pb(C_2H_3O_2)_2,3H_2O$	0.0189 "
Magnesia	MgO	0.0020 "
Magnesium carbonate	$MgCO_3$	0.0042 "
Nitrogen	N	0.0014 "
Potassium acetate*	$KC_2H_3O_2$	0.0098 "
" bicarbonate	$KHCO_3$	0.0100 "
" bitartrate *	$KHC_4H_4O_6$	0.0188 "
" carbonate	K_2CO_3	0.0069 "
" citrate *	$K_3C_6H_5O_7,H_2O$	0.0106 "
" hydroxide	KOH	0.0056 "
" and sodium tartrate	$KNaC_4H_4O_6,4H_2O$	0.0141 "
Sodium acetate	$NaC_2H_3O_2,3H_2O$	0.0136 "
" benzoate *	$NaC_7H_5O_2$	0.0144 "
" bicarbonate	$NaHCO_3$	0.0084 "
" borate	$Na_2B_4O_7,10H_2O$	0.0191 "
" carbonate	Na_2CO_3	0.0053 "
" "	$Na_2CO_3,10H_2O$	0.0143 "
" hydroxide	$NaOH$	0.0040 "
" salicylate *	$NaC_7H_5O_3$	0.0160 "

No. 241. DECINORMAL SODIUM HYDROXIDE SOLUTION. One cc. is equivalent to

Acid, acetic	$H,C_2H_3O_2$	0.0060 gram.
" boric	H_3BO_3	0.0062 "
" citric	$H_3C_6H_5O_7,H_2O$	0.0070 "
" hydrobromic	$HBBr$	0.0081 "
" hydrochloric	HCl	0.00365 "
" hydriodic	HI	0.0128 "
" lactic	$HC_3H_5O_3$	0.0090 "



No. 142. DECINORMAL IODINE SOLUTION. One cc. is equivalent to

Arsenious oxide	As_2O_3	0.00495 gram.
Potassium sulphite	$K_2SO_3,2H_2O$	0.0097 "
Sodium bisulphite	$NaHSO_3$	0.0052 "
" sulphite,	$Na_2SO_3,7H_2O$	0.0126 "
" thiosulphate	$Na_2S_2O_3,5H_2O$	0.0248 "
Sulphur dioxide	SO_2	0.0032 "
Sulphurous acid	H_2SO_3	0.0041 "

No. 245. DECINORMAL SODIUM THIOSULPHATE SOLUTION. One cc. is equivalent to

Bromine	Br	0.0080 gram.
Chlorine	Cl	0.00355 "
Iodine	I	0.01266 "
Iron (in ferric salts)	Fe	0.0056 "

No. 230. DECINORMAL SILVER NITRATE SOLUTION.* One cc. is equivalent to

Ammonium bromide	NH_4Br	0.0098 gram.
" chloride	NH_4Cl	0.00535 "
Chlorine	Cl	0.00355 "
Cyanogen	$(CN)_2$	0.0052 "
Hydrocyanic acid	HCN with indicator	0.0027 "
" "	HCN { to formation of precipitate }	0.0054 "
Hydrobromic acid	HBr	0.0080 "
Potassium bromide	KBr	0.0119 "
" chloride	KCl	0.00745 "
" cyanide	KCN with indicator	0.0065 "
" "	KCN { to formation of precipitate }	0.0130 "
Sodium bromide	NaBr	0.0103 "
" chloride	NaCl	0.00585 "

No. 201. DECINORMAL POTASSIUM BICHROMATE SOLUTION.† One cc. is equivalent to

Ferrous carbonate	$FeCO_3$	0.0116 gram.
Ferric oxide	Fe_2O_3	0.0080 "
Ferrous oxide	FeO	0.0072 "
" sulphate	$FeSO_4$	0.0152 "
" "	$FeSO_4,7H_2O$	0.0278 "
Iron (ferrous)	Fe	0.0056 "

No. 220. DECINORMAL POTASSIUM PERMANGANATE SOLUTION. One cc. is equivalent to

Oxalic acid $H_2C_2O_4,2H_2O$ 0.0063 gram,

and to same weights for iron salts as given under N/10 $K_2Cr_2O_7$.

* Use potassium chromate solution as an indicator, or add till precipitate appears.

† Use a freshly prepared solution of potassium ferricyanide as an indicator, applying a drop of titrated solution to a drop of indicator on a white surface.

The following table from Talbot * shows the reactions of the common indicators used in acidimetry:

Indicator.	Reaction with Acids.	Reaction with Alkalies.	Use with Carbonic Acid in Cold Solution.	Use with Carbonic Acid in Hot Solution.	Use with Ammonium Salts.	Use with Organic Acid.
Litmus.....	Red	Blue	Unreliable	Reliable	Reliable	Reliable
Methyl orange..	Pink	Yellow	Reliable	Unreliable	Reliable	Unreliable
Phenolphthalein..	Colorless	Pink	Unreliable	Reliable	Unreliable	Reliable
Lacmoid.....	Purple-red	Blue	Unreliable	Reliable	Reliable	Unreliable (?)
Cochineal.....	Purple-red	Blue	Reliable	Reliable	Reliable	Unreliable
Rosolic acid.....	Yellow	Pink	Unreliable	Reliable	Unreliable	Unreliable†
Alizarine.....	Yellow	Red	Unreliable	Reliable	Reliable	Reliable

* Talbot, Quantitative Analysis, page 75.

† Reliable with oxalic acid.

REFERENCES ON LABORATORY EQUIPMENT, REAGENTS, ETC.

ADRIANCE, J. S. Laboratory Calculations. New York, 1897.

ATKINSON, E. Suggestions for the Establishment of Food Laboratories. U. S. Dept. of Agric., Off. of Exp. Sta., Bul. 17.

COHN, A. J. Tests and Reagents, Chemical and Microscopical, known by their Authors' Names. New York, 1903.

KENWOOD, H. R. Public Health Laboratory Work. The Hygienic Laboratory. Philadelphia, 1893.

KRAUCH, C. Testing of Chemical Reagents for Purity. London, 1903.

LUNGE, G., and HURTER, F. Alkali-maker's Handbook. London, 1891.

MAYRHOFER, J. Instrumente und Apparate zur Nahrungsmittel Untersuchung. Leipzig, 1894.

SCHNEIDER, A. Reagents and Reactions known by the Names of their Authors. 1897.

SUTTON, F. Volumetric Analysis. 8th Ed. Philadelphia, 1900.

THORPE, T. E. Dictionary of Applied Chemistry. London, 1894.



CHAPTER III.

FOOD, ITS FUNCTIONS, PROXIMATE COMPONENTS, AND NUTRITIVE VALUE.

Nature and General Composition.—Food is that which, when eaten, serves by digestion and absorption to support the functions and powers of the body, by building up the material necessary for its growth and by supplying its wastes. The raw materials that constitute our food-supply are not all available for nourishment, but often contain a proportion of inedible or refuse matter, which it is customary to remove before eating, such as the bones of fish and meat, the shells of clams and oysters, eggshells, the bran of cereals, and the skins, stones, and seeds of fruits and vegetables. The proximate components which make up the edible portion of food include in general water, fat, various nitrogenous bodies collectively known as *protein*, carbohydrates, organic acids, and mineral matter. Of these, water is hardly to be considered as a nutrient, though it plays an important part in nearly all foods as a diluent and solvent. The fat, protein, and carbohydrates all contribute in varying degree to the supply of fuel for the production of heat and energy. Besides this universal function, the fats and the carbohydrates serve especially to furnish fatty tissue in the body, while the protein is the chief source of muscular tissue.

Liebig's classification of foods into *nitrogenous*, or flesh formers, and *non-nitrogeneous*, or heat generators, is now no longer accepted as strictly logical, in view of the well-known fact that the nitrogenous materials, besides building up the body, aid in supplying the wastes and yielding energy, while the non-nitrogenous aid in furnishing tissue growth in addition to serving as fuel. Indeed protein is itself convertible into fats and carbohydrates within the body by peculiarly complex processes.

THE FAT OF FOOD.—Fats are the glycerides of the fatty acids, the characteristics of the various edible fats and oils being treated of under

their appropriate headings elsewhere. Fat in human food is supplied by milk and its products, by the adipose tissue of meat, and in slight extent by the oil of cereals and by the edible table oils. The term "ether extract" is sometimes used synonymously with fat, though other substances than fat are, when present, extracted by ether, such as chlorophyl and other coloring matters, lecithin, alkaloids, etc.

PROTEIN.—This term covers a wide variety of nitrogenous bodies, and in one form or another is found in nearly all foods, both animal and vegetable. The terms "proteids" and "albuminoids" are used generically by some chemists as synonymous with "protein" to include all nitrogenous bodies in food. The proteids and albuminoids strictly speaking are, however, subdivisions of the nitrogenous compounds, though in reality they are undoubtedly by far the most important from the standpoint of nutrition.

Protein available for food is supplied chiefly by the flesh of meat and fish, by milk, cheese, and eggs, and to a smaller degree by the grains and vegetables, especially the legumes.

CLASSIFICATION OF NITROGENOUS BODIES.—Nitrogenous compounds occurring in food are for convenience divided as follows: A. Proteids, B. Albuminoids, C. Amido-Compounds, D. Alkaloids, E. Nitrates, F. Ammonia, and G. Lecithin. The subdivision of the proteids and albuminoids as here given follows in the main the plan of Watts.*

A. PROTEIDS.—These are highly complex and usually amorphous compounds of carbon, hydrogen, nitrogen, oxygen, and sulphur, capable of conversion by certain enzymes and digestive juices through a process of hydration into proteoses and peptones. The latter after absorption in the body are reconverted into proteids. No marked distinction exists between animal and vegetable proteids. All proteids are insoluble in

a proteid solution produces a white precipitate, which becomes brick-red on heating. Sodium chloride prevents the reaction. Various organic salts are precipitated by Millon's reagent, but the precipitate does not turn red on heating.

Biuret Reaction.—If a solution of a proteid in dilute sulphuric acid be made alkaline with potassium or sodium hydroxide and *very dilute* copper sulphate be added, a reddish to violet coloration is produced, similar to that formed if biuret* be treated in the same way; hence the name. An excess of copper sulphate should be avoided lest its color obscure that of the reaction.

Piotrowski's Reaction.—Dilute copper sulphate added to a moderately strong solution of a proteid forms a precipitate of albuminate of copper.

Phosphotungstic Acid Reaction.—According to Stutzer, all proteid matters in aqueous, alkaline, or acid solution are precipitated by saturation with a strongly acid solution of sodium phosphotungstate. For use as a reagent, dissolve the latter in sulphuric, acetic, or phosphoric acid.

(a) **ALBUMINS.**—These bodies are soluble in cold water and in saturated solutions of sodium chloride. They are precipitated by saturating with ammonium sulphate, and coagulated by heat.

Occurrence.—*In Animal Foods:* Eggs, milk.

In Vegetable Foods: Wheat, rye, and potatoes.

Qualitative Tests.—All the general tests for proteids apply to albumins.

Coagulation.—Animal albumins usually coagulate at about 75°; vegetable albumins at about 65°.

Miscellaneous Reactions.—Dilute acids precipitate albumin with the aid of heat. Nitrate of mercury (in dilute nitric acid) precipitates albumins from their solutions; also Mayer's solution acidified with acetic acid.

(b) **GLOBULINS.**—These bodies are insoluble in water, soluble in dilute saline solutions, but insoluble in concentrated solutions of sodium chloride and magnesium or ammonium sulphate. They are coagulated by heat.

Occurrence.—*In Animal Foods:* As myosin in muscle of meat; as vitellin in yolk of egg.

In Vegetable Foods: As vegetable vitellin in cereals and legumes.

Qualitative Tests.—Globulins are precipitated from their solution with

---

* Biuret is the substance formed by heating urea to 160° according to the following reaction:

$$\underset{\text{Urea}}{2CON_2H_4} = \underset{\text{Biuret}}{C_2O_2N_3H_5} + \underset{\text{Ammonia}}{NH_3}$$

saturated magnesium sulphate or sodium chloride. Albumins are not thus precipitated.

(c) **ALBUMINATES.** — These are compounds formed by the action of weak acids and alkalies on globulins or albumins. They are insoluble in water and in natural solutions containing no salt, but soluble in acids and alkalies. They are not coagulated by heat.

Occurrence.—*In Animal Foods:* Casein of milk.

In Vegetable Foods: As legumin in peas and beans; as congluten in almonds.

Qualitative Tests. — Albuminates like globulins are precipitated by saturation with neutral salts.

If sulphuric acid is added to a solution of an albuminate in excess of acetic acid, a violet coloration and a slight fluorescence are produced.

(d) **PROTEOSES.**—These are bodies formed from proteids by the action of proteolytic ferments, being intermediate products in the hydration of the proteids to peptones. They might be considered as semi-digested proteids. They may be artificially formed by heating various proteids with water, mineral acids, or steam. They are not coagulated by heat, and are insoluble in alcohol.

Occurrence.—*In Animal Foods:* In sour milk and ripened cheese, as caseose.

In Vegetable Foods: In wheat flour, as abrus.

Subdivision of the Proteoses.—According to the proteids from which they are derived the proteoses are subdivided into *albumose*, from albumin, *globulose*, from globulin, *vitellose*, from vitellin, *caseose*, from casein, etc.

Three varieties of albumose are as follows: *proto-albumose*, soluble in water (both cold and hot) and in dilute salt solutions, but precipitated by



are soluble in water, insoluble in alcohol, not coagulated by heat, and not precipitated by nitric acid.

Hemi-peptones are those which are capable of being further decomposed into simpler bodies, such as leucine and tyrosine, by prolonged action of pancreatic juice.

Anti-peptones are those incapable of further decomposition and which do not respond to Millon's reaction.

Occurrence.—Both forms of peptones, being diffusible in animal membranes, are found in meat, but are not present in milk. They are not found in plants.

Qualitative Tests.—Besides giving the biuret reaction, peptones are precipitated from their solution by tannic acid, picric acid, phosphomolybdic acid, and by sodium phosphotungstate acidified by acetic, phosphoric, or sulphuric acid.

Peptones are the only soluble proteids not precipitated by saturation with ammonium sulphate. The following table is due to Halliburton:*

Variety of Proteid.	Hot and Cold Water.	Hot and Cold Saline Solutions, e.g., 10% NaCl.	Saturation with NaCl or $MgSO_4$.	Saturation with $(NH_4)_2SO_4$	Nitric Acid.	Copper Sulphate.	Copper Sulphate and Caustic Potash.
Proto-albumose	Soluble	Soluble	Precipitated	Precipitated	Precipitated in cold; precipitate dissolves with heat and reappears on cooling	Precipitated	Rose-red color (biuret reaction)
Hetero-albumose	Insoluble; i.e. precipitated by dialysis from saline solutions	Soluble; partly precipitated, but not coagulated on heating to 65° C.	Precipitated	Precipitated	Ditto	Precipitated	Ditto
Deutero-albumose	Soluble	Soluble	Not precipitated	Precipitated	This reaction occurs only in presence of excess of salt	Not precipitated	Ditto
Peptone	Soluble	Soluble	Not precipitated	Not precipitated	Not precipitated	Not precipitated	Ditto

(f) **INSOLUBLE PROTEIDS.**—These are insoluble in water and dilute acids, but soluble with heating in strong mineral acids. The proteids of this class most commonly met with are fibrin and myosin in animal foods, and gluten in wheat.

B. ALBUMINOIDS.—The albuminoids constitute a class of nitrogenous bodies closely allied to and in many respects resembling the proteids, including the following products found in animal foods:

* Chemical Physiology and Pathology, page 131.

(a) **Collagen,** composing the fibers of connective tissue, is prepared from finely divided tendon by first soaking out the soluble proteids with cold water, then removing the mucin by soaking in lime water, and finally washing with water and dilute acetic acid. By boiling with water or subjecting to steam under pressure collagen is converted into

(b) **Gelatin,** which is prepared also from bones by boiling. Insoluble in cold, but soluble in hot water. When the hot water solution cools it forms a jelly, if one per cent or more of gelatin is present in the solution. By prolonged boiling the gelatinizing power is lost. Aqueous solutions of gelatin are strongly lævo-rotary.

Qualitative Tests.—Gelatin is precipitated from its solution by mercuric chloride, picric acid, or tannic acid. It is readily distinguished from the proteids in that it is not precipitated from its solution by lead acetate, nor by most of the metallic salts that throw down the proteids.

(c) **Mucin.**—This is the principal ingredient of the ground substance of connective tissues in meat, and is also the chief component of mucus. It is precipitated by lead acetate, but by no other metallic salt. It responds to the xanthoproteic and Millon's reactions.

(d) **Nuclein** is the chief component of the nuclei of cells, and contains much phosphorus. It occurs in the yolk of eggs and in milk. It is also present in yeast and in tea leaves.

(e) **Chondrin.**—This is obtained from cartilage by long boiling. It is a transparent, gelatin-like substance, and in aqueous solution is precipitated by the same reagents that precipitate gelatin.

(f) **Elastin** is the substance which forms the elastic fibers of connective tissue. It is very insoluble, and in fact no solvent is known which does not decompose it. It is digestible by both pepsin and trypsin.

C. **AMIDES, AMIDO-ACIDS, AND ALLIED PRODUCTS.**—Under this



after having removed the albumin by coagulation (by boiling) and by filtration.*

Asparagin when heated with alkalies forms ammonia, and with acids forms ammonium salts. Freshly prepared copper hydroxide is dissolved by an aqueous solution of asparagin by the aid of heat. If sections of vegetable tissues containing asparagin are placed in alcohol, crystals of asparagin are formed in such a manner as to be detected under the microscope.†

Closely allied to the amides are the flesh bases of meat, chief among which are *creatin* ($C_4H_9N_3O_2$), *creatinin* ($C_4H_7N_3O$), derived from creatin by the action of mineral acids and existing in some fish, *carnin* ($C_7H_8N_4O_3$), and *xanthin* ($C_5H_4N_4O_2$).

D. ALKALOIDAL NITROGEN. — Alkaloids do not naturally occur in foods, with the exception of tea, coffee, and kola-nuts, which contain caffeine, and cocoa, which contains theobromine.

E. NITROGEN AS NITRATES.—Foods in their natural condition rarely contain nitrates. Meats cured with saltpetre furnish the most common instance of nitrates in food. Nitrates are tested for by extracting the sample with water, and treating the extract with ferrous sulphate and sulphuric acid in the usual manner.

F. NITROGEN AS AMMONIA. — Ammonia occurs very sparingly in food, unless the latter has undergone some form of decomposition. In ripened cheese and in sour milk one sometimes finds it in minute quantities. Its presence is tested for by distilling the finely divided sample in water free from ammonia, and testing the distillate with Nessler's reagent.

G. LECITHIN.—This substance ($C_{44}H_{90}NPO_9$) is a phosphorized fat, and forms a part of the cell material in certain animal and vegetable foods. It is found in considerable quantity in the yolk of egg, and, in traces, in cereals, peas, and beans. It is a yellowish-white solid, soluble in ether and alcohol. Treated with water it swells up, forming an opalescent solution or emulsion, from which it is precipitated by salts of the alkali metals.

THE CARBOHYDRATES AND THEIR CLASSIFICATION.—The carbohydrates supplied by food are milk sugar and the various sugars, starches, and gums from plant juices, cereals, fruits, and vegetables. Carbohydrates are generally understood as being compounds of carbon, hydrogen, and oxygen, the last two elements being present in the proportion in

* Zeits. für analytische Chemie, 22, page 325.

† Wiley. Principles of Agric'l Analysis, Vol. III. p. 427.

which they occur in water. They are divided into three main classes, as follows:

A. The Glucose Group, or Monosaccharids ($C_6H_{12}O_6$), including dextrose, levulose, and galactose.

B. The Cane Sugar Group, or Disaccharids ($C_{12}H_{22}O_{11}$), including cane sugar, milk sugar, and maltose.

C. The Cellulose Group ($C_6H_{10}O_5$), including starch, cellulose, dextrin, gums, etc.

Closely allied to the carbohydrates, if not actually belonging to them, are *inosite* ($C_6H_{12}O_6$), occurring in muscular tissue, and *pectose*, found in green fruits and vegetables.

THE ORGANIC ACIDS.—These acids are minor though important constituents of foods. From their conversion into carbonates within the body, they are useful in furnishing the proper degree of alkalinity to the blood and to the various other fluids, besides being of particular value as appetizers. They exist in foods both in the free state and as salts. Acetic acid is supplied by vinegar; lactic acid by milk, fresh meat, and beer; citric, malic, and tartaric acids by the fruits.

MINERAL OR INORGANIC MATERIALS. — These substances occur in food in the form of chlorides, phosphates, and sulphates of sodium, potassium, calcium, magnesium, and iron, and are furnished by common salt, as well as by nearly all animal and vegetable foods. The inorganic salts are necessary to supply material for the teeth and bones, besides having an important place in the blood and in the cellular structure of the entire body.

FUEL VALUE OF FOOD.—In order to express the capacity of foods for yielding heat or energy to the body, the term fuel value is commonly used. By the fuel value of a food material is meant the amount of heat expressed in calories equivalent to the energy which we assume the body could obtain



Fig. 13, consists of a water-tight, cylindrical, platinum lined, steel bomb, adapted to hold in a capsule the substance whose heat is to be determined, and containing also oxygen under pressure. This bomb is immersed in water contained in a metal cylinder, which is in turn placed inside of concentric cylinders containing alternately air and water. The heat for igniting the substance is supplied by the electric current passing through wires to the interior of the bomb and acting upon a cleverly devised mechanism therein. The heat developed by the ignition is measured by

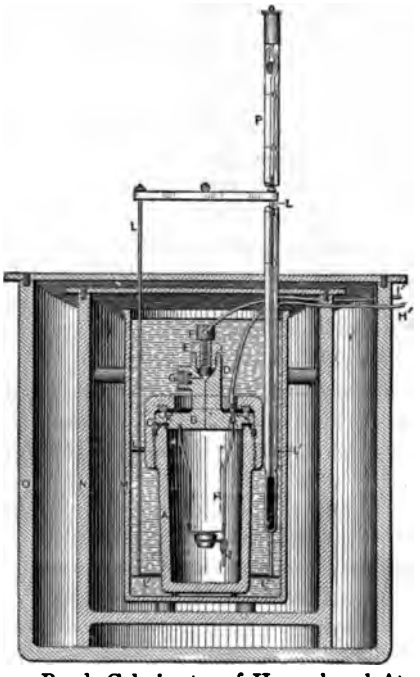


FIG. 13.—Bomb Calorimeter of Hempel and Atwater

the rise in temperature of the water surrounding the bomb, as indicated by a very delicate thermometer graduated to hundredths of a degree, certain corrections being made, as, for instance, for the heat absorbed by the metal of the apparatus. A mechanical stirrer serves to equalize the temperature of the water surrounding the bomb.

Calculation of Fuel Value.—By reason of its great expense the calorimeter is beyond the reach of many laboratories, and on this account the expression of fuel values by calculation is the most common method employed. For this the factors of Rubner are generally used, in accordance with which the amount of energy in one gram of each of the three principal

classes of nutrients are, for carbohydrates 4.1, for protein 4.1, and for fats 9.3. Expressed in pounds, each pound of carbohydrate or protein has a fuel value of 1860 calories, while each pound of fat has a fuel value of 4220 calories.

REFERENCES ON DIETETICS AND ECONOMY OF FOOD.

ARMSBY, H. P. The Principles of Animal Nutrition. New York, 1903.
ATWATER, W. O. Dietaries in Public Institutions. Yearbook of U. S. Dept. of Agric., 1901, page 393.
—— Food and Diet. Yearbook of U. S. Dept. of Agric., 1894, page 357.
—— Principles of Nutrition and Nutritive Value of Food. Farmer's Bulletin, 142.
BELLOWS, A. J. The Philosophy of Eating. Boston, 1867.
BURNET, R. W. Foods and Dietaries. Phil., 1893.
BRYANT, A. P. Some Results of Dietary Studies in the United States. Yearbook of U. S. Dept. of Agric., 1898, page 439.
HALLIBURTON, W. D. Text-book of Chemical Physiology and Pathology. London, 1891.
HAMMARSTEN, O. A Text-book of Physiological Chemistry. New York, 1898.
HUTCHINSON, ROBT. Food and the Principles of Dietetics. New York, 1901.
JAFFA, M. E. The Study of Human Foods and Practical Dietetics. Cal. Exp. Sta., Bul. 110.
KNIGHT, J. Food and its Functions. London, 1895.
NEUMEISTER, A. Lehrbuch der physiologische Chemie. 1897.
PAVY, T. W. A Treatise on Food and Dietetics. London, 1874.
RICHARDS, E. H. The Cost of Living as Modified by Sanitary Science. New York, 1900.
—— The Cost of Food: A Study in Dietaries. New York, 1901.
SIMPSON, H. Choice of Food. Manchester, England, 1889. Lectures.
STROHMER, F. Die Ernährung des Menschen.
THOMPSON, W. G. Practical Dietetics. New York, 1895.
TOWNSHEND, S. H. The Relation of Food to Health. St. Louis, 1897.
TRUE, A. C. and MILNER, R. D. Development of Nutrition Investigations of the

Bul. 31. Dietary Studies at the University of Missouri in 1895, and Data Relating to Bread and Meat Consumption in Missouri. By H. B. Gibson, S. Calvert, and D. W. May, with comments by W. O. Atwater and C. D. Woods. Pp. 24.

Bul. 32. Dietary Studies at Purdue University, Lafayette, Ind., in 1895. By W. E. Stone, with comments by W. O. Atwater and C. D. Woods. Pp. 28.

Bul. 35. Food and Nutrition Investigations in New Jersey in 1895 and 1896. By E. B. Voorhees. Pp. 40.

Bul. 37. Dietary Studies at the Maine State College in 1895. By W. H. Jordan. Pp. 57.

Bul. 38. Dietary Studies with Reference to the Food of the Negro in Alabama in 1895 and 1896. Conducted with the cooperation of the Tuskegee Normal and Industrial Institute and the Agricultural and Mechanical College of Alabama. Reported by W. O. Atwater and C. D. Woods. Pp. 69.

Bul. 40. Dietary Studies in New Mexico in 1895. By A. Goss. Pp. 23.

Bul. 44. Report of Preliminary Investigations on the Metabolism of Nitrogen and Carbon in the Human Organism with a Respiration Calorimeter of Special Construction. By W. O. Atwater, C. D. Woods, and F. G. Benedict. Pp. 64.

Bul. 45. A Digest of Metabolism Experiments in which the Balance of Income and Outgo was Determined. By W. O. Atwater and C. F. Langworthy. Pp. 434.

Bul. 46. Dietary Studies in New York City in 1895 and 1896. By W. O. Atwater and C. D. Woods. Pp. 117.

Bul. 52. Nutrition Investigations in Pittsburg, Pa., 1894–1896. By Isabel Bevier. Pp. 48.

Bul. 53. Nutrition Investigations at the University of Tennessee in 1896 and 1897. By C. E. Wait. Pp. 46.

Bul. 54. Nutrition Investigations in New Mexico in 1897. By A. Goss. Pp. 20.

Bul. 55. Dietary Studies in Chicago in 1895 and 1896. Conducted with the cooperation of Jane Addams and Caroline L. Hunt, of Hull House. Reported by W. O. Atwater and A. P. Bryant. Pp. 76.

Bul. 56. History and Present Status of Instruction in Cooking in the Public Schools of New York City. Reported by Mrs. Louise E. Hogan, with an introduction by A. C. True, Ph.D. Pp. 70.

Bul. 63. Description of a New Respiration Calorimeter and Experiments on the Conservation of Energy in the Human Body. By W. O. Atwater and E. B. Rosa. Pp. 94.

Bul. 68. A Description of Some Chinese Vegetable Food Materials and their Nutritive and Economic Value. By W. C. Blasdale. Pp. 48.

Bul. 69. Experiments on the Metabolism of Matter and Energy in the Human Body. By W. O. Atwater and F. G. Benedict, with the cooperation of A. W. Smith and A. P. Bryant. Pp. 112.

Bul. 71. Dietary Studies of Negroes in Eastern Virginia in 1897 and 1898. By H. B. Frissell and Isabel Bevier. Pp. 45.

Bul. 75. Dietary Studies of University Boat Crews. By W. O. Atwater and A. P. Bryant. Pp. 72.

Bul. 84. Nutrition Investigations at the California Agricultural Experiment Station, 1896–1898. By M. E. Jaffa. Pp. 39.

Bul. 85. A Report of Investigations on the Digestibility and Nutritive Value of Bread. By C. D. Woods and L. H. Merrill. Pp. 51.

Bul. 89. Experiments on the Effect of Muscular Work upon the Digestibility of Food and the Metabolism of Nitrogen. Conducted at the University of Tennessee, 1897–1899. By C. E. Wait. Pp. 77.

Bul. 91. Nutrition Investigations at the University of Illinois, North Dakota Agricultural College, and Lake Erie College, Ohio, 1896–1900. By H. S. Grindley and J. L. Sammis, E. F. Ladd, and Isabel Bevier and Elizabeth C. Sprague. Pp. 42.

Bul. 98. The Effect of Severe and Prolonged Muscular Work on Food Consumption, Digestion, and Metabolism, by W. O. Atwater and H. C. Sherman, and the Mechanical Work and Efficiency of Bicyclers, by R. C. Carpenter. Pp. 67.

Bul. 107. Nutrition Investigations among Fruitarians and Chinese at the California Agricultural Experiment Station, 1899–1901. By M. E. Jaffa. Pp. 43.

Bul. 109. Experiments on the Metabolism of Matter and Energy in the Human Body, 1898–1900. By W. O. Atwater and F. G. Benedict, with the cooperation of A. P. Bryant, A. W. Smith, and J. F. Snell. Pp. 147.

Bul. 116. Dietary Studies in New York City in 1896 and 1897. By W. O. Atwater and A. P. Bryant. Pp. 83.

Bul. 117. Experiments on the Effect of Muscular Work upon the Digestibility of Food and the Metabolism of Nitrogen. Conducted at the University of Tennessee, 1899–1900. By C. E. Wait. Pp. 43.

Bul. 121. Experiments on the Metabolism of Nitrogen, Sulphur, and Phosphorus in the Human Organism. By H. C. Sherman. Pp. 47.



CHAPTER IV.

GENERAL ANALYTICAL METHODS.

Extent of a Proximate Chemical Analysis.—For purposes of studying the proximate composition of food for its dietetic value, it is nearly always necessary to make determinations of moisture, ash, fat, total nitrogen, and carbohydrates (when present), as well as of the fuel value. In some cases it may be desirable to proceed further, to make an analysis of the ash, for instance, to separate, at least into classes, the various nitrogenous bodies, especially in flesh foods, and perhaps to subdivide the starch, sugar, gums, and cellulose or crude fiber that make up the carbohydrates in the case of cereals.

An analysis is considered complete whenever the purpose for which the examination has been made has been accomplished, and on that purpose depends solely the extent to which the various compounds present shall be subdivided and determined. Such a subdivision may be extended almost indefinitely. For example, a milk analysis may consist simply in the determination of the total solids and (by difference) the water. Again, it may be desirable to divide the milk solids into fat and solids not fat, and in some cases to carry the subdivision still farther and separate the solids not fat into casein, albumin, milk sugar, and ash.

Determinations of one or more of the proximate components natural to food are frequently of great service in proving their purity or freedom from adulteration. For the latter purpose, especially in such foods as milk, vinegar, oils, and fats, the determination of specific gravity is also an important factor. Special methods of a peculiar nature are often necessary in the examination of particular foods, and such methods will be treated subsequently under the appropriate headings. In the present chapter only such general methods as are applicable to a large variety of cases will be discussed.

Expression of Results of a Proximate Analysis.—However complete the division of the various proximate compounds or classes of compounds

which the analyst sees fit to make, the results of his various determinations in a proximate analysis are expected to aggregate about 100%. If every determination be directly made, the result will rarely be exactly 100, but the precision of the work is apt to be judged by its approach to 100.

It is often the custom to determine certain compounds or classes of compounds by difference. Thus in cereals the moisture, the proteids, the fat, and the ash may be determined by the regular analytical methods, and by subtracting their sum from 100 the difference may be expressed as "nitrogen-free extract" or carbohydrates. It has long been customary in food analysis to calculate the protein by multiplying the total nitrogen by the factor 6.25, and on this basis analyses of thousands of animal and vegetable foods have been made. While the figure thus obtained is an arbitrary one, being at best but a rough approximation of the amount of protein present, yet for many reasons there is much to commend this practice of reporting results. In the first place, in most cases it actually does approach the truth. Again, the nitrogenous ingredients of many foods are so numerous and varied, that for the every-day study of dietaries and food values it would be well-nigh impossible with our present knowledge to subdivide these compounds with any degree of accuracy, and especially with uniformity between different chemists, to say nothing of the time involved.

From the fact that so many valuable analyses have already been expressed on the basis of $N \times 6.25$ for protein, the advantage of comparison with the results thus recorded would seem to be in itself a good reason for continuing the practice, especially until a factor that gives better average results can be adopted. By recording the actual nitrogen found



Preparation of the Sample.—It is at the outset of the utmost importance in all cases that a strictly representative portion of the food to be examined should be submitted to analysis. All refuse matter, such as bones, shells, bran, skins, etc., are removed as completely as possible from the edible portion and discarded.

If the composition of the entire mass cannot be made homogeneous throughout, it may be best to select from various portions in making up the sample for analysis, in order to represent as fair an average of the whole as possible.

Finally the sample, if solid or semi-solid, should be divided as finely as possible, by chopping, shredding, pulping, grinding, or pulverizing according to its nature and consistency.

For disintegrating such substances as vegetables and meats for analysis, the common household rotary chopping-machine is admirably adapted. For pulverizing cereals, tea, coffee, whole spices, and the like, the mortar and pestle may be used, or a rotary disk mill or spice-grinder.

Specific Gravity or Density of Liquids.—Where formerly it was customary to compare the density of liquids with that of water at 4° C. (its maximum density) it is now more common to refer to water at 15.5° C. or 60° F., making the determination at that temperature. A common form of expressing the temperature of the determination and the temperature of the standard volume of water with which that of the substance is to be compared, is the employment of a fraction, the numerator of which expresses the temperature of the determination and the denominator that of the standard volume of water, as $\frac{15.5^\circ}{4^\circ}$, $\frac{15.5^\circ}{15.5^\circ}$, $\frac{100^\circ}{15.5^\circ}$, $\frac{4^\circ}{4^\circ}$ C.* When extreme accuracy in the determination of density is required, the pycnometer or Sprengel tube should be employed.

The Hydrometer.—This instrument furnishes the most convenient and ready means of determining the density of liquids where extreme nicety is not required. If well made and carefully adjusted, the hydrometer may be depended on to three decimal places, but before relying on its accuracy, it should be tested by comparison with a standard instrument, or with the pycnometer.

The liquid whose density is to be determined is contained in a jar whose inner diameter should be at least $\frac{3}{8}''$ larger than that of the spindle-

* Unless otherwise stated, all specific gravities in this volume are assumed to be expressed on the basis of $\frac{15.5^\circ}{15.5^\circ}$

bulb, and the temperature of the liquid should be exactly 15.5° when the reading is taken.

For best results for use with liquids of varying densities, the laboratory should be furnished with a set of finely graduated hydrometers, each limited to a restricted part of the scale, together with a universal hydrometer coarsely graduated, covering the entire range, to show by preliminary test which of the special instruments should be used.

A convenient set of seven such hydrometers are graduated as follows: 0.700–0.850, 0.850–1.000, 1.000–1.200, 1.200–1.400, 1.400–1.600, 1.600–1.800, 1.800–2.000, while the universal hydrometer has a scale extending from 0.700 to 2.000. Another less delicate set of three only has one for liquids lighter than water and two for heavier liquids. Some instruments have thermometers in the stem. Others require a separate thermometer.

The Westphal Balance (Fig. 14).—This instrument consists of a scale-beam fulcrumed upon a bracket, which in turn is upheld by a supporting pillar. The scale-beam is graduated into ten equal divisions. From a hook on the outer end of the beam hangs a glass plummet provided with a delicate thermometer, the beam being so adjusted that when the dry plummet hangs in the air, the beam is in exact equilibrium, i.e., perfectly horizontal as shown by the indicator on its inner end. If the large rider be placed on the same hook as the plummet and the latter immersed in distilled water of the standard temperature at which the determinations are to be made (say 15.5° C.), the scale-beam should again be in equilibrium if the instrument is accurately adjusted. As commonly made, the weight of the plummet including the platinum wire to which it is attached amounts to 15 grams, and the displacement of its volume to 5 grams of distilled water at 15.5° C., the normal temperature on which the determinations are based. Thus the unit (or largest) rider



stored, the third and the fourth riders successively. If it happens that two riders should occupy the same position on the beam, the smaller is suspended from the larger.

If the liquid is heavier than water, the method employed is the same except that one of the largest or unit riders is in this case always hung from the hook which supports the plummet, while the others cross the



FIG. 14.—The Westphal Balance.

beam at the proper points. If carefully made and adjusted, the Westphal balance is capable of considerable accuracy.

A delicate analytical balance can be used in place of the less carefully adjusted Westphal instrument, by hanging the Westphal plummet from one of the scale-hooks of the same, and employing a fixed support for the glass jar that holds the liquid in which the plummet is to be immersed. The support is so arranged that the scale-pan below it can move freely without coming in contact with it. This arrangement is shown in Fig. 15.

The Pycnometer, or Specific-gravity Bottle. — Fig. 16 shows the two

forms of pycnometer commonly made. The plain form has a ground-glass stopper with a capillary passage through it, the other has a fine thermometer connected with the stopper and a capillary side tube provided with a ground hollow cap. Both are made in different sizes to hold respectively 10, 25, 50, and 100 grams of distilled water at the standard

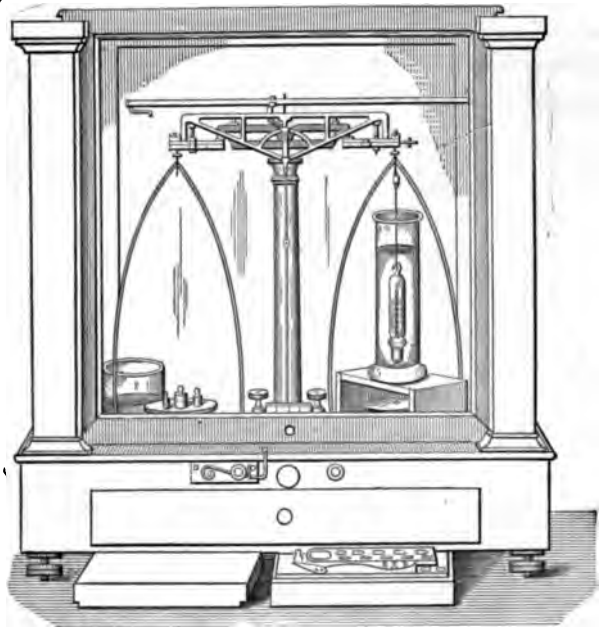


FIG. 15.—The Analytical Balance Arranged for Determining Specific Gravity with the Westphal Plummet.

temperature. It is convenient to have a counterweight for each pycnom-

on the balance, when the temperature has reached the standard (say 15.5° C.), being careful to wipe off the excess of liquid that exudes from the capillary due to expansion. The net weight of the liquid is divided by that of the same volume of distilled water, previously ascertained under the same conditions at the same temperature, the result being the density of the liquid.

The pycnometer with thermometer attachment is obviously susceptible of a greater degree of accuracy than the other form, since the temperature of the liquid, even though 15.5° C. at the start, soon rises.

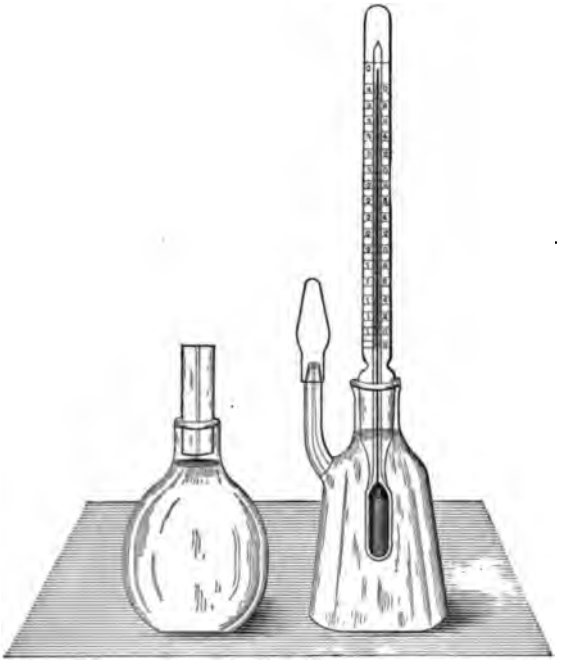


FIG. 16.—Types of Pycnometer.

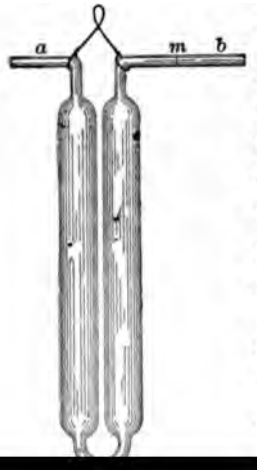
The writer prefers to use the pycnometer provided with the thermometer, but without the hollow cap that covers the capillary side tube, unless liquids like strong acids are to be operated on, that might otherwise injure the balance. By keeping the liquid to be tested for some time in a refrigerator, it acquires a temperature of from 10 to 12° C. It is then transferred in the regular manner to the pycnometer and the thermometer-stopper inserted (but not the hollow cap) and the bottle wiped dry. There is ample time to adjust the balance-weights with extreme care while the temperature of the liquid is rising, leisurely wiping off

at intervals with a soft towel the excess that exudes from the capillary tube, the final weight of the dry bottle and contents being made at the exact temperature of 15.5° C.

In taking the tare or adjusting the counterweight of a specific-gravity bottle, the latter should be perfectly clean and dry. It had best be rinsed first with water, then with alcohol, and finally with ether, all traces of the latter being removed by a current of dry air, or otherwise, before weighing.

In making successive determinations of density of a number of different liquids with the same pycnometer, it is sufficient to rinse the bottle once with a little of the liquid to be tested before making each determination, when the various liquids are miscible. When the liquids are immiscible, the bottle should be carefully cleaned in the manner described in the previous paragraph before making each test.

The Sprengel Tube.—The Sprengel tube is a variety of pycnometer useful when only a small quantity of the liquid to be tested is available. It is susceptible of great accuracy. It consists of a U-shaped tube (Fig. 17), each branch of which terminates in a horizontal capillary tube bent outward. One of the capillaries, *b*, has a mark *m* thereon and has an inner diameter of about 0.5 mm. The diameter of the other capillary, *a*, should not exceed 0.25 mm. The liquid at room temperature is aspirated into the tube so as to fill it completely, the end *b* being dipped in the liquid while suction is applied at the end *a*. The tube is then placed in a beaker of water kept at the standard temperature, the beaker being of such size that the capillary ends rest on the edge. The temperature of the liquid in the tube may be assumed to be constant



Determination of Moisture.—The moisture is usually calculated from the weight of dry residue left after driving out all the water by evaporation from a weighed portion of the sample, using generally from 1 to 10 grams in a tared dish. Some substances readily part with their water at 100°; others, again, require a much higher temperature or an extremely long heating. In general the highest possible degree of heat should be employed that will not affect the other constituents. Certain saccharine products should theoretically be dried at a temperature not exceeding 70° on account of the dehydration of some of the sugars at higher temperatures. On the contrary, where readily decomposable organic matter is known to be absent and the character of the substance will permit, it is sometimes possible to employ temperatures considerably above 100° for quick drying.

It is not always safe to assume that water is the only substance evaporated on drying. Thus spices and other products containing essential oils give off appreciable quantities of these oils when dried at 100°.

As it is rarely possible to attain a temperature higher than 98° in the water-oven, a gas-heated air-oven of the general type of that described on page 21, with ready means for controlling the temperature, is best for general moisture determinations in the food laboratory.

Platinum dishes like those described on page 97 are admirable for nearly all moisture determinations, but thin dishes of porcelain, glass, or metal may be used. Thin liquids and air-dry, powdered substances may usually be weighed directly in the dish and dried, without the use of an absorbent.

With very moist substances containing much cellulose as well as water, it is often advantageous to weigh into the dish and allow to simmer for a long time on the water-bath, before drying to constant weight in the air-oven at higher temperature.

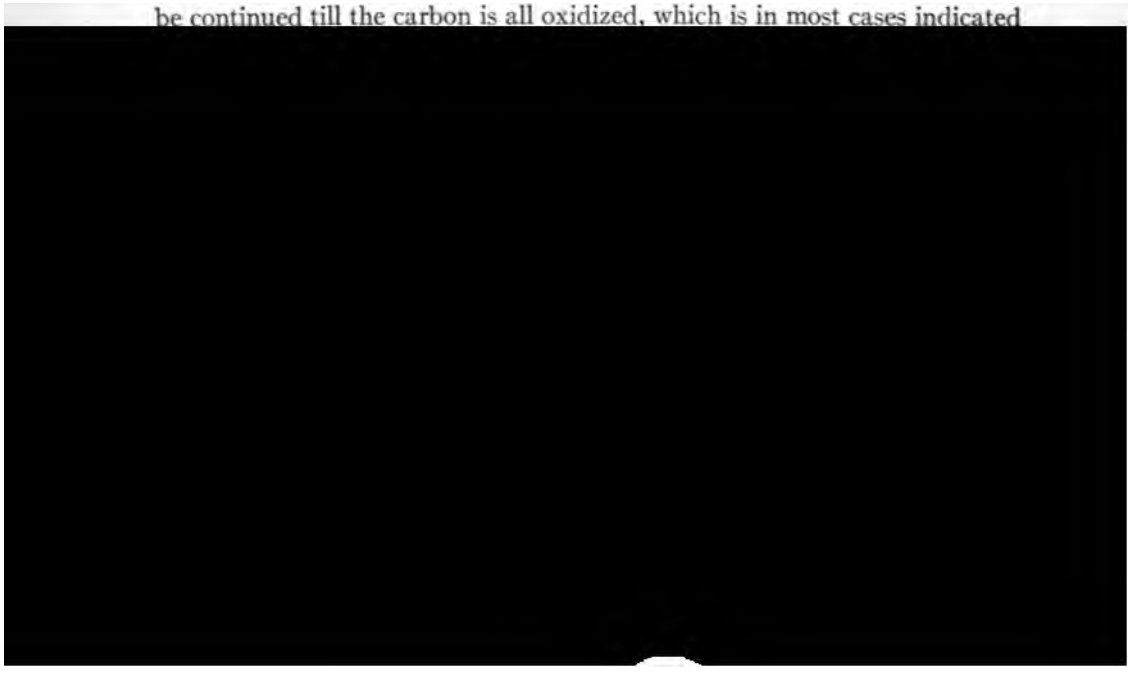
Viscous substances should generally be spread over finely divided asbestos fiber, or pieces of pumice stone, or sea sand, which should be previously ignited and weighed with the dish, the object being to divide up the weighed portion as finely as possible for its better exposure to the heated air in the drying-oven.

Determination of Ash.—For determining the percentage of the ash or mineral matter, it is often convenient to use the portion previously weighed out and dried in obtaining the moisture, the dry residue after the second weighing being in such cases burnt in the original dish over a low flame. Or, if desired, a fresh portion of the original substance may

be air-dried or subjected to a preliminary drying in the water-bath and then burnt, taking care that there is no loss by sputtering or otherwise.

Platinum dishes will be found much the most convenient in all cases where they may be safely used. In general the shallow flat dishes described on page 97 are preferable. Where lead or tin compounds are present, or when sulphides, sulphates, or phosphates are to be burnt with reducing agents, platinum is sure to be attacked and porcelain dishes or crucibles should be used instead. For the ordinary care of platinum dishes it would be well to know that platinum is attacked at ordinary temperatures by free chlorine and bromine, and, when ignited, by free sulphur, phosphorus, arsenic, and iodine. Platinum dishes are liable to injury also when used for the ignition of sulphates, sulphides, and phosphates with reducing substances, or with metals present that are reduced in fusion, such as mercury, bismuth, tin, lead, zinc, antimony, etc.

The degree of heat employed in ashing should be the lowest possible to insure complete oxidation of the carbon, so as to avoid driving off certain volatile salts that are sometimes present and that would be lost if the heat were too high. In general a dull red heat is preferable, the ash being usually best obtained by the direct action of the gas flame on the open dish containing the sample, the dish being suspended on a triangle or other support above the flame. It is rarely necessary to employ the muffle in obtaining the ash of food products. There are some substances, however, that would injure a platinum dish if ignited over the free flame which may be ashed with safety in a muffle. Thus the cereals like whole wheat and barley have been found to ruin platinum dishes when ignited therein directly over the flame, doubtless due to the phosphates present, but they may safely be ignited in platinum in the muffle. Heating should be continued till the carbon is all oxidized, which is in most cases indicated



trated sulphuric acid, when the presence of sulphates in the ash is not objectionable.

Methods for the detection and determination of the various ash ingredients are in general the familiar processes of the analytical chemist and will not be here considered. Such cases, however, as are peculiar to certain foods, like the metallic impurities that occur in canned, bottled, and preserved foods under certain conditions, will be considered in their appropriate place.

Extraction with Volatile Solvents.—Wherever it is necessary to exhaust a substance of its ether-soluble or alcohol-soluble ingredients, some form of continuous extraction apparatus is employed with advantage.

The Soxhlet Extractor.—This apparatus, or one of its modifications is most commonly employed for continuous extraction. Fig. 18 shows the simplest form of the Soxhlet extractor, consisting essentially of a wide tube, *A*, provided with the side siphon *a*, a condenser, *B*, and a wide-mouthed flask, *C*, all connected together in the manner illustrated, either by soft, accurately fitting corks or by ground joints, or by mercury-sealed connections. Care in either case should be taken to have the joints perfectly tight, so as to avoid loss by leakage. The construction is such that the substance to be extracted, which is contained in the tube *A*, is subjected to successive treatment with freshly distilled portions of the solvent. The vapor from the solvent, boiling in the flask *C*, passes up through the side tube *a'* into the cold condenser *B*, where it is again reduced to liquid and falls drop by drop upon the substance to be extracted, which is confined in a suitable porous receptacle or perforated vessel in the tube *A*. The substance is thus allowed to macerate in the solvent till the level of the latter reaches the top of the siphon, when all of the solvent in the tube drains off into the flask *C*, carrying with it whatever it dissolves. The operation is at once repeated, the substance

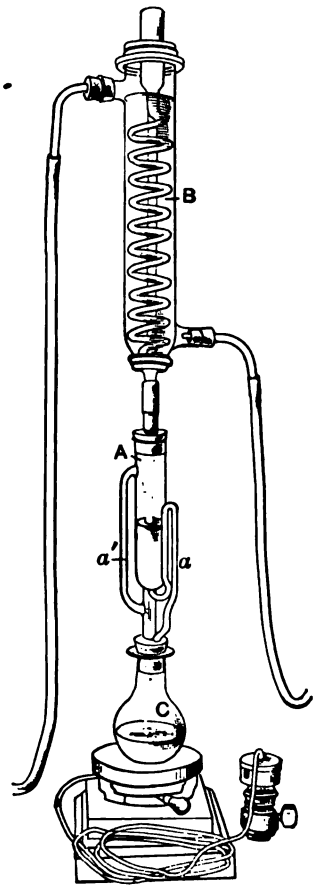
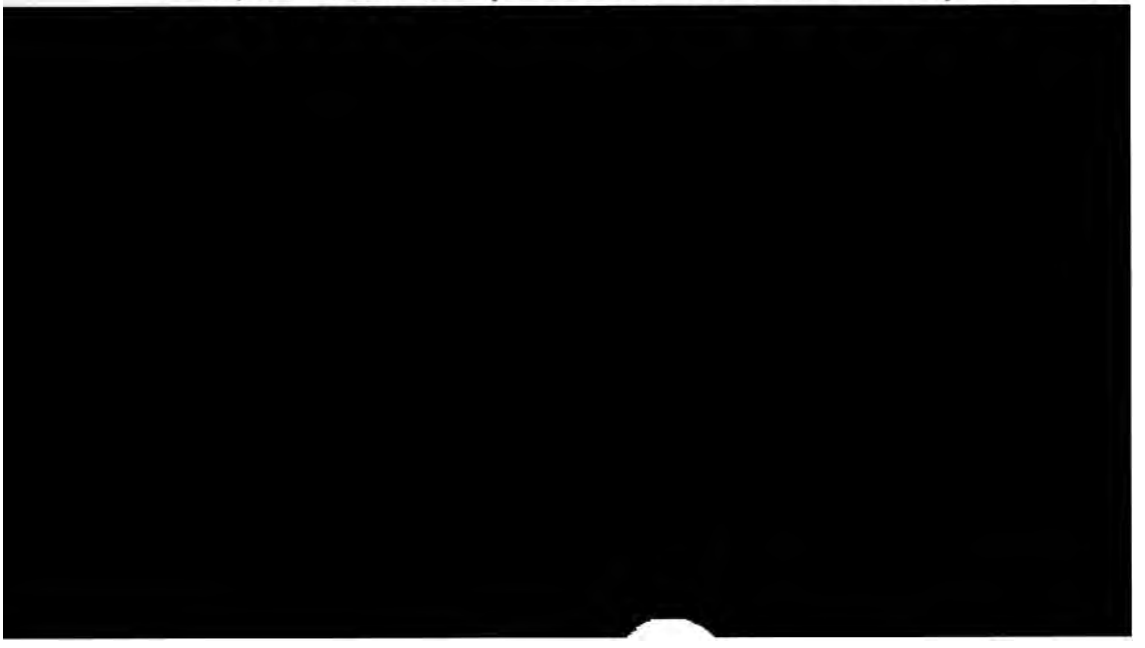


FIG. 18.—The Soxhlet Extractor with Electric Heater.

being subjected to successive extractions with freshly distilled portions of the solvent, which leaves behind in the flask *C* whatever it dissolves. This operation of continuous extraction, when the conditions are right, goes on indefinitely without attention.

The weighed portion of the sample to be extracted (from 2 to 5 grams) is first deprived of its moisture by drying, if free from volatile oil, and then transferred to the bottom of the tube *A*. There are various methods of doing this. If the substance is a fluid or semi-fluid like milk, it may be taken up on an absorption-coil of fat-free filter-paper and dried (see page 99), the dried coil being transferred to the tube *A*. Or the sample may be weighed into a very thin glass shell (Hoffmeister's Schälchen) in which it is dried, after which the shell is wrapped in bibulous paper, crushed between the fingers into small bits, and the whole, in the form of a small packet, is placed in the tube *A*. Or, again, the material, if in the form of an air-dried powder, may be weighed in a tared platinum dish or watch-glass and transferred by a brush into a partly folded wrapper of filter-paper, the ends of which are afterwards closed in by folding to form a packet, which is first dried thoroughly in the oven and then placed in the tube *A*. The fat-free porous shells made by Schleicher & Schull, in various sizes to fit the Soxhlet tubes, form convenient receptacles for the extraction of dry substances. The sample may in most cases be directly weighed into one of these shells after taking its tare, and the drying and extraction carried out at once.

Extraction with Ether.—In taking the so-called ether extract, sometimes reckoned as fat, the solvent employed is either ethyl ether or the cheaper petroleum ether. Whichever reagent is employed, certain precautions are necessary for the purity of the reagent. If ethyl ether is used, it should be entirely freed from moisture and alcohol by first



be considered. A convenient still for fractionating such substances as petroleum ether is shown in Fig. 19.

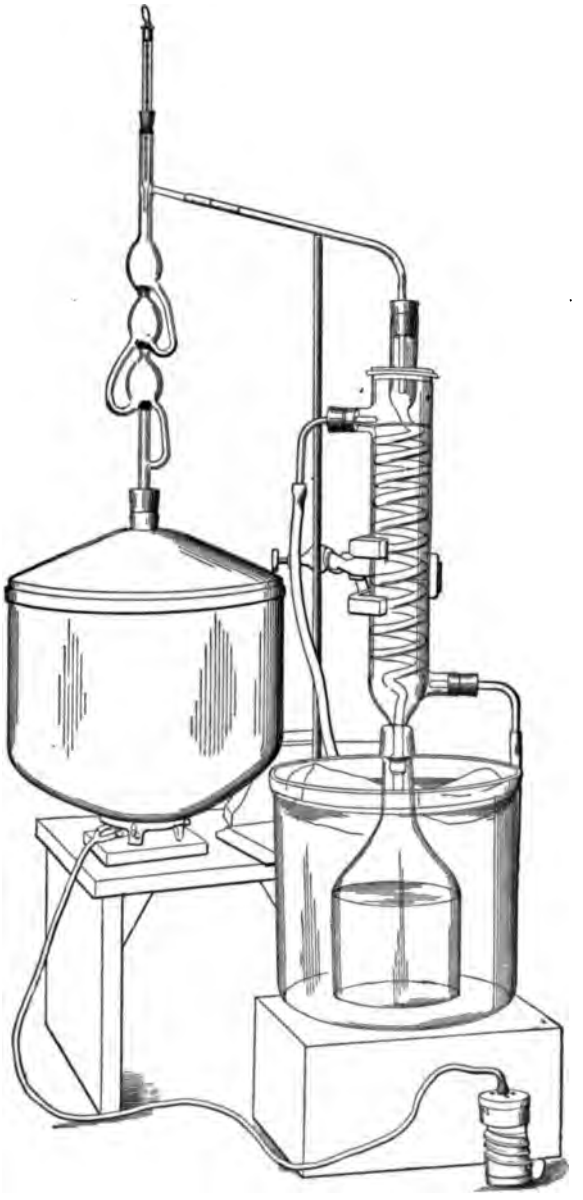


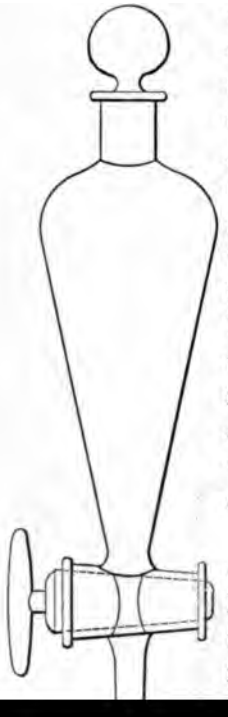
FIG. 19.—Fractionating-still, Arranged for Petroleum Ether.

Method of Conducting the Extraction.—The flask *C*, Fig. 18, is first thoroughly cleaned and dried and then weighed, after which enough of

the solvent reagent is poured into it to last through the period of the extraction, and the parts of the apparatus are connected.

The heater employed should be a water-bath, or, as shown in Fig. 18, an electric stove, which may be provided with a fractional rheostat for varying the amount of heat.

The degree of ebullition is so regulated as to allow the solvent to saturate the sample and siphon over into the flask *C* from six to twelve times an hour, the extraction being continued from two to six hours, or until all the ether-soluble material has been removed. Care should be taken also that the rate of boiling and the rate of condensation are so regulated that no appreciable loss of reagent occurs during the extraction. A strong smell of ether perceptible at the top of the condenser indicates a loss. The solvent is recovered at the end of the extraction by disconnecting the weighing-flask at a time when nearly all of the solvent is in the part *A* and before it is ready to siphon over. The weighing-flask is then freed from all traces of the solvent by drying first on the water-bath and then in the oven, after which it is cooled in the desiccator and weighed, the difference between this and the first weighing representing the weight of the fat or ether extract.



Extraction with Immiscible Solvents. — It is frequently necessary to dissolve out a substance from a liquid by shaking it with an immiscible solvent, as, for example, in the extraction of certain preservatives from aqueous or acid solutions with ether, petroleum

out through the top. If troublesome emulsions form when shaken, they may frequently be broken up by adding an excess of the solvent and again very gently shaking, or by careful manipulation with a stirring-rod. If the solvent is ether, and an obstinate emulsion forms, it may frequently be broken by the addition of chloroform. Such a mixture of ether and chloroform sinks to the bottom and may be drawn off as in the case of chloroform alone. Ether or chloroform emulsions that refuse to yield to either of the above methods may often be broken by the aid of a centrifuge.

Determination of Nitrogen by Moist Combustion.—In thus determining nitrogen, the organic matter is first decomposed by digestion with an oxidizer, the carbon and hydrogen being driven off in gaseous form as carbon dioxide and water respectively, while the nitrogen is converted into an ammonium salt, from which free ammonia (NH_3) is later liberated by making alkaline. The ammonia is then distilled into an acid solution of known value and calculated by titrating the excess of acid.

The Kjeldahl process of moist combustion, involving the use of mercury or mercuric oxide in oxidizing and of potassium sulphide in distilling, has been largely superseded by the simpler Gunning method, which is better adapted for general food work.

The method as described in its simplest form is not applicable in the presence of nitrates, but the latter occur very rarely in foods in appreciable amounts. The ordinary Gunning method is not well adapted to the determination of nitrogen in pepper, on account of the difficulty with which piperin undergoes decomposition. In this case nitrogen is best determined by the Gunning-Arnold method, page 334.

The Gunning Method.—*Reagents:*

Standard alkali solution, N/10 NaOH.
Pulverized potassium sulphate.
Sulphuric acid, concentrated.
Sodium hydroxide, saturated solution.
Standard acid solution, N/10 H_2SO_4 or HCl.
An indicator, cochineal.
Granulated zinc.

The digestion and distillation are preferably carried out in the same flask, which should be pear-shaped with round bottom and made of hard, moderately thick, well-annealed glass. A convenient size has the following dimensions: length 29 cm., maximum diameter 10 cm., tapering gradu-

ally to a long neck, which near the end is 28 mm. in diameter with a flaring edge. Its capacity is about 550 cc.

If desired, the digestion may be conducted in a smaller hard-glass flask of about 250 cc. capacity and of the same shape as the above, and the distillation in an ordinary round-bottomed flask of 500 cc. capacity.

Introduce from 0.5 to 3.5 grams of the sample into the digestion-flask with 10 grams of potassium sulphate and from 15 to 25 cc. of concentrated sulphuric acid. The flask is inclined over the flame and heated gently for a few minutes below the boiling-point of the acid till the frothing has ceased, after which the heat is gradually increased till the acid boils, and the boiling is continued till the contents have become practically colorless or at least of a pale straw color. Wire gauze is interposed between the flask and flame. A funnel placed in the neck of the flask acts as a return-flow condenser.

The contents of the flask are then cooled, and, if the digestion has been conducted in the larger flask suitable also for distilling, as above recommended, 200 cc. of water are added and sufficient strong sodium hydroxide to make the contents strongly alkaline, using phenolphthalein as an indicator. If a separate flask is used for the distillation, the contents of the digestion-flask are transferred thereto with the water and the alkali added. A few pieces of granulated zinc should also be introduced, which by the evolution of gas prevents bumping and the sucking back of the distillate. The flask is then well shaken and connected with the condenser, the bottom of which is provided with an adapter, dipping below the surface of the standard hydrochloric or sulphuric acid, a measured quantity of which is contained in the receiving-flask. The distillation is then continued till all the ammonia has passed over into the acid, this

Modification of Gunning Method to include Nitrogen of Nitrates.—In addition to the reagents used in the simpler Gunning method, sodium thiosulphate and salicylic acid are required.

A mixture of salicylic and sulphuric acids is made in the proportion of 30 cc. of concentrated sulphuric to 1 gram of salicylic. From 30 to 35 cc. of

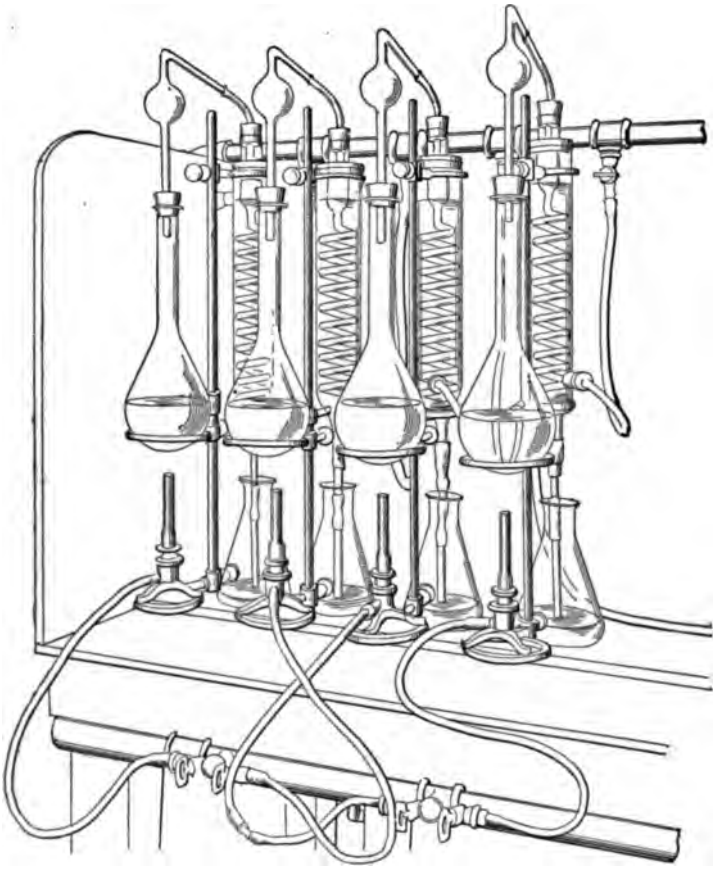


FIG. 21.—Bank of Stills for Nitrogen Determination by Gunning Process.

the mixture are added to the 0.5 to 3.5 grams of the substance in the digestion-flask, the flask is well shaken and allowed to stand a few minutes, occasionally shaking. Then 5 grams of sodium thiosulphate are added, and 10 grams of potassium sulphate, after which the heat is applied, at first very gently and afterwards increasing slowly till the frothing has ceased. The heating is then continued till the contents have been boiled practically colorless. From this point on, proceed as in the Gunning method.

Determination of Free Ammonia.—A weighed quantity of the finely divided sample, treated with ammonia-free water and made alkaline with barium or magnesium carbonate, is distilled into a measured quantity of standard acid (tenth-normal hydrochloric or sulphuric acid) and the amount of ammonia determined by titration.

Determination of Amido-nitrogen.*—In the absence of ammonia, or after the removal of the ammonia as described in the preceding section, the sample is boiled for an hour with 5% hydrochloric or sulphuric acid, which converts the amido-compounds into ammonium salts (chloride or sulphate). Assuming asparagin to be the amido-compound acted upon, the reaction is as follows:

$$\underset{\text{Asparagin}}{2C_4H_8N_2O_3} + H_2SO_4 + 2H_2O = \underset{\text{Ammonium sulphate}}{(NH_4)_2SO_4} + \underset{\text{Aspartic acid}}{2C_4H_7NO_4}.$$

Exactly neutralize the free acid with sodium carbonate, add magnesia (free from carbonate), and distil into standard tenth-normal acid. The ammonia is determined by titration in the usual manner, and its nitrogen represents half of the nitrogen contained in the amido-compound, which it is customary to calculate as asparagin.

Determination of the Various Carbohydrates.—Under title of "Cereals" in Chapter IX are given in detail methods for separation and determination of sugar, dextrin, crude fiber, etc.

POISONED FOODS.—Such metallic impurities as are present in food products incidental to their preparation, or as adulterants, will be considered under title of the foods liable to such adulteration.

The detection of highly toxic substances in food, such as arsenic, mercuric chloride, the alkaloids, and other organic poisons that do not occur in food naturally or accidentally, and are present not as adulterants properly

Detection of Arsenic.—In testing most food substances for arsenic, it is usually unnecessary to entirely destroy the organic matter, but whenever possible the substance under examination should, by treatment with concentrated nitric and sulphuric acids, be brought into the form of a dry char, which may readily be divided finely by the action of a pestle in a mortar. In this condition the arsenic, which by the process has been oxidized to arsenic acid, may be completely dissolved by continual treatment with boiling water. The hot-water solution containing the extract of the powdered char is then cooled, filtered, and submitted to the Marsh apparatus.

For preliminary treatment of liquids or semi-liquid substances, proceed as directed under arsenic in beer, page 591.

In treating substances like meats, vegetables, and the like, follow in general the directions of Chittenden and Donaldson * for organic tissues, the proportions of acid, etc., being varied to suit special conditions. Heat in a porcelain dish 100 grams of the finely divided substances with 23 cc. of pure concentrated nitric acid at a temperature between 150° and 160° C., stirring occasionally with a glass rod. After the substance has taken on a deep yellow or orange color, remove the dish from the heat, add 3 cc. of pure concentrated sulphuric acid, and stir while the nitrous fumes are given off. The operator should wear a rubber glove to protect the hands. Again heat to about 180° and add while hot, drop by drop, 8 cc. of pure concentrated nitric acid, stirring during the addition of the acid. Then heat at 200° till sulphuric acid fumes come off and a dry carbonaceous mass remains.

This is then pulverized and exhausted with boiling water, and the aqueous solution, when cold, submitted to the Marsh test.

The Marsh Apparatus and its Operation.—Fig. 22 shows a simple form of Marsh apparatus applicable for this work. The generator is provided with a doubly perforated rubber stopper, containing the usual delivery-tube and the entrance-tube. The latter has for convenience a 60° funnel at the top, into which the filter-paper can be folded and the solution containing the extract filtered directly into the generator. A chloride of calcium drying-tube is interposed between the generator and the capillary tube, the latter being drawn from hard arsenic-free tubing.

The apparatus is operated in the usual manner, using arsenic-free

* American Chem. Journal, II. No. 4; Chem. News, Jan. 1881, p. 21.

granulated zinc and dilute sulphuric acid. After running the current of hydrogen long enough through the heated tube to prove the absence of arsenic in the reagents or apparatus, the aqueous solution of the char is poured into the moistened filter at the top of the funnel-tube and allowed to filter slowly into the generator. The length of time necessary to deposit in the capillary tube all the arsenic in the sample, or to prove the absence of arsenic, varies with the conditions, but in general if no darkening of

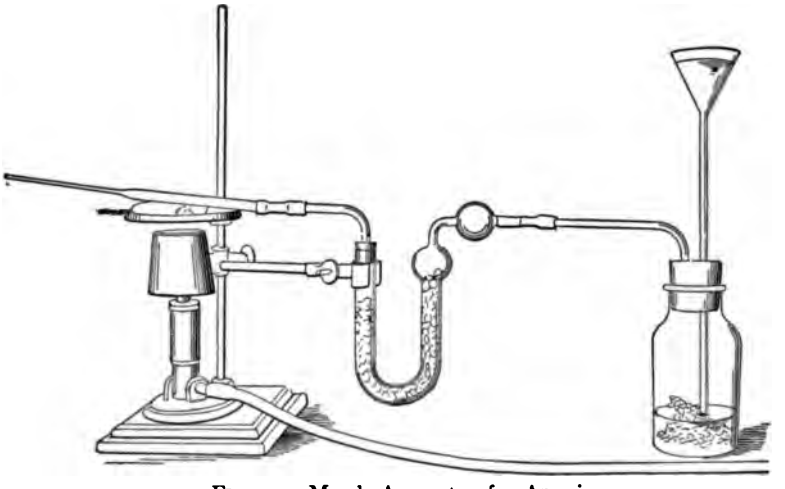


FIG. 22.—Marsh Apparatus for Arsenic.

the tube occurs after an hour, the sample may be considered free from arsenic.

*Estimation of Arsenic.**—With the aid of an assay balance sensitive to 0.00001 gram, it is possible to weigh with accuracy an arsenic mirror



is then cooled and again weighed on the assay balance, the difference in weight corresponding to the metallic arsenic.

If the amount of arsenic is small, it may be estimated by Sanger's method,* which consists in comparing the mirror in the capillary with a series of standard mirrors, made by using varying measured amounts of a standard arsenious oxide solution. This solution is prepared by dissolving 1 gram of pure arsenious oxide (As_2O_3) in water with the aid of arsenic-free sodium carbonate, and, after acidification with dilute sulphuric acid, making up to a liter. Ten cc. of this solution are measured out carefully and made up to a liter with water, the strength of the dilute solution being 0.01 mgr. to 1 cc. One cc., 2 cc., 3 cc., etc., of this second or dilute solution are separately measured into the Marsh apparatus to give mirrors corresponding to the same number of hundredth-milligrams.

REFERENCES TO GENERAL FOOD ANALYSIS.

ALLEN, A. H. Commercial Organic Analysis. Philadelphia, 1898.

BATTERSHALL, J. P. Food Adulteration and its Detection. New York, 1887.

BELL, JAS. The Analysis and Adulteration of Foods, Pts. I and II. London, 1881.

BLYTH, A. W. and M. W. Foods, their Composition and Analysis. New York, 1903.

BÖHMER, C. Die Kraftfuttermittel, ihre Rohstoffe, Herstellung, Zusammensetzung, etc. Berlin, 1903.

BUJARD, A., and BAIER, E. Hilfsbuch für Nahrungsmittel Chemiker. Berlin, 1894.

BURCKER, E. Traité des Falsifications et Alterations des Substances alimentaires et des Boissons. Paris, 1892.

DIETZSCH, O. Die Wichtigsten Nahrungsmittel und Getränke. Zurich, 1884.

ELSNER, F. Praxis der Nahrungsmittel Chemiker. Leipzig, 1880.

EPHRAIM, J. Originalarbeiten über Analyse der Nahrungsmittel. Leipzig, 1894.

GIRARD, C., et DUPRÉ, A. Analyse des Matières alimentaires et Recherche des leurs Falsifications. Paris, 1894.

HANAUSEK, T. F. Die Nahrungs- und Genussmittel aus dem Pflanzenreiche. 1884.

HASSALL, A. H. Food, its Adulterations and the Methods for their Detection. London, 1874.

KÖNIG, J. Chemische Zusammensetzung der menschlichen Nahrungs- und Genussmittel. Berlin, 1903.

LEACH, A. E. Food: Methods of Inspection and Analysis. Article in Reference Handbook of the Medical Sciences, Vol. 3, pages 180–183.

LEFFMANN, H., and BEAM, W. Select Methods of Food Analysis. Philadelphia, 1901.

POLIN et LABIT. Examen des Aliments suspects. Paris, 1892.

RÖTTGER, H. Kurzes Lehrbuch der Nahrungsmittel Chemie. Leipzig, 1903.

RUPP, G. Die Untersuchung von Nahrungsmitteln, Genussmitteln und Gebrauchsgegenständen. 1900.

* Proc. Acad. of Arts and Sciences, XXVI. (1891) p. 24.

THOMS, H., und GILG, E. Einführung in die praktische Nahrungsmittel-Chemie. Leipzig, 1899.
VILLIERS, A., et COLLIN, E. Traité des Alterations et Falsifications des Substances alimentaires. Paris, 1900.
WIESSNER. Die Rohstoffe des Pflanzenreiches. Leipzig, 1900.
WILEY, H. W. Principles and Practice of Agricultural Analysis. Vol. III. Agricultural Products. Chem. Pub. Co., Easton, Pa., 1897.

The Analyst. London, 1877 et seq.
Revue International des Falsifications. Amsterdam, 1888 et seq.
Vierteljahresschrift der Chemie der Nahrungs- und Genussmittels. Berlin, 1884 et seq. (Discontinued 1897.)
Zeitschrift für Untersuchung der Nahrungs- und Genussmittel. 1898 et seq.
Vereinbarungen zur Untersuchung und Beurtheilung von Nahrungs- und Genussmitteln. Berlin, 1897.

Also the following bulletins of the Bureau of Chemistry, U. S. Deptartment of Agriculture:
Bulletin 13, Parts 1–10. Food and Food Adulterants. 1887–1902.
Bulletin 46. Methods of Analysis adopted by the A. O. A. C. 1899.
Bulletin 65. Provisional Methods for the Analysis of Foods, adopted by the A. O. A. C. Nov. 14–16, 1901. 1902.



CHAPTER V.

THE MICROSCOPE IN FOOD ANALYSIS.

Microscopical vs. Chemical Analysis.—A very important means of identification of adulterants in many classes of food products is furnished by the microscope, which in many cases affords more actual information as to the purity of food than can be obtained by a chemical analysis. This is especially true of coffee, cocoa, and the spices, wherein the microscope serves to reveal not only the nature of the adulterants, but also not infrequently the approximate amount of foreign matter present. In the case of the cereal and leguminous starches so commonly employed as adulterants, a microscopical examination is of paramount importance.

The chemical constants of many of the adulterants of coffee and the spices do not always differ sufficiently from those of the pure foods in which they appear to be distinguished therefrom with accuracy and confidence by a chemical analysis alone. On the other hand, one who is familiar with the appearance under the microscope of the pure foods and of the starches and various ground substances used as adulterants, can, with certainty, identify very minute quantities of these materials, when present, with the same ease that one can recognize megascopically the most familiar objects about him.

A chemical test may, for example, indicate the presence of starch, but it cannot reveal the particular kind of starch. The microscope will at once show whether the starch present is wheat or corn or potato or arrowroot, since these starches differ almost as much in microscopical appearance as do the physical characteristics of the grains or tubers from which they are obtained. Again, by a chemical analysis an abnormal amount of crude fiber may show the presence of a woody adulterant, but only the microscope will enable one to decide whether the impurity consists of sawdust or ground cocoanut shells. Not only in such instances as these is the microscopical examination of greater importance

than a chemical analysis in establishing the purity of the food, but it is at the same time a much quicker guide.

The Technique of Food Microscopy.—The recognition of adulterants by the microscope is not difficult, and by no means involves the labor and skill necessary for almost all other kinds of histological work. In the examination of cocoa, coffee, tea, and the spices for adulteration, a careful study of the powdered substance in temporary water mounting will in most cases prove sufficient to familiarize the food analyst with their characteristics under the microscope, and it is not absolutely necessary for him to familiarize himself with the details of section cutting, dissecting, or permanent mounting unless he so desires. The treatment in detail of these latter branches of vegetable histology is beyond the scope of the present work. For full information along these lines the reader is referred especially to such works as those of Behrens * and of Zimmerman,† together with the list of references on page 86.

Standards for Comparison.—For standards the analyst should provide himself with as complete a set as possible of the various materials to be examined, taking care that their absolute purity is established. Wherever possible, he should grind the sample himself from carefully selected whole goods. These, together with samples of the starches and other adulterants, all of known purity, should be contained in small vials carefully stoppered and plainly labeled, arranged alphabetically or in some equally convenient manner in the desk or table on which the microscope is commonly used. The adulterants included in this set of standards should be not only those which experience has shown most liable to be employed, but any which, by reason of their character, might in the analyst's opinion be used under certain conditions.



A stand of the so-called continental type (having the horseshoe base) is preferable. A square stage is rather more convenient than the circular form, and the jointed pillar possesses advantages over the rigid variety in ease of manipulation that are certainly worth considering.

The smooth working of both the coarse and fine adjustments should not be lost sight of. If the microscope is to be used exclusively for food work, a substage condenser is unnecessary, hence the construction of the substage may be very simple, unless bacteriological work is to be done as



FIG. 23.—Continental Type of Microscope.

well. The stand should be so constructed as to allow the analyst to readily bring into place the parts of a polarizing apparatus and as quickly to remove them.

A nose-piece, while not indispensable, is a great convenience for the quick transfer of objectives. A double nose-piece carrying two objectives is ample for routine food work.

The Optical Parts are by far the most important, and should be of superior quality, though not necessarily of the most expensive makers. The food analyst should have at least two objectives, one for high- and one for low-power work, and preferably two oculars.

For the routine examination of powdered food substances the writer prefers a ⅓-inch objective, used with a medium ocular, the combination

giving a magnification of from 240 to 330 diameters, according to the ocular employed. For a low-power objective the $\frac{3}{4}$-inch is a convenient grade, which, in connection with a low-power eyepiece, is well adapted for the examination of butter and lard, and for use with the polariscope.

The Micro-polariscope.—This accessory is useful in the identification of starches and other ingredients, and for ascertaining whether or not fats have been crystallized. The most convenient form is that in which the polarizer is held below the stage, while the analyzer is applied above the objective, between it and the tube. This form permits the use of any desired ocular.



FIG. 24.—Polarizer and Analyzer for the Microscope.

A common form of construction is one in which the substage is adapted to carry interchangeably the diaphragm tube and the polarizer. If the polariscope is much used, it becomes desirable to provide means for quickly changing the polarizer and diaphragm tube below the stage, and for moving the analyzer in and out of place above the objective. Winton * has devised a microscope-stand with this in view, especially adapted to the needs of

Other useful accessories are a mechanical stage, a pair of fine tweezers, knives, scissors, and, if sections are to be cut, a plano-concave razor.

Preparation of Vegetable Food Products for Microscopical Examination.—The ground spices and cocoas of commerce are usually of the requisite fineness for direct examination without further treatment. Coffee, chocolate, starches, and similar products should be ground in a mortar fine enough to pass through a sieve with from 60 to 80 meshes to the inch.

A small portion of the powdered sample is taken up on the tip of a clean, dry knife-blade, and placed on the microscope-slide. By means of a medicine-dropper a drop of distilled water is applied, and the wetted

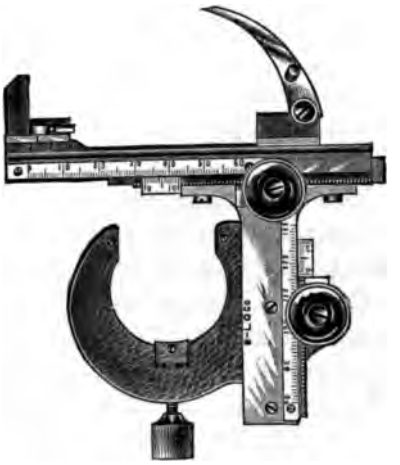


FIG. 25.—Mechanical Stage for Microscope.

powder is then rubbed out under the cover-glass between the thumb and finger to the proper fineness.

The water-mounted slide thus prepared, while useful only for temporary purposes, has proved to be best adapted to the analyst's requirements for routine microscopical examination of powdered food products for adulteration, partly because water is the best medium in most cases for showing up the structural characteristics of these substances and their adulterants, and partly because it serves best for the "rubbing out" process between thumb and finger under the cover-glass, whereby the sample is brought to the requisite degree of fineness.

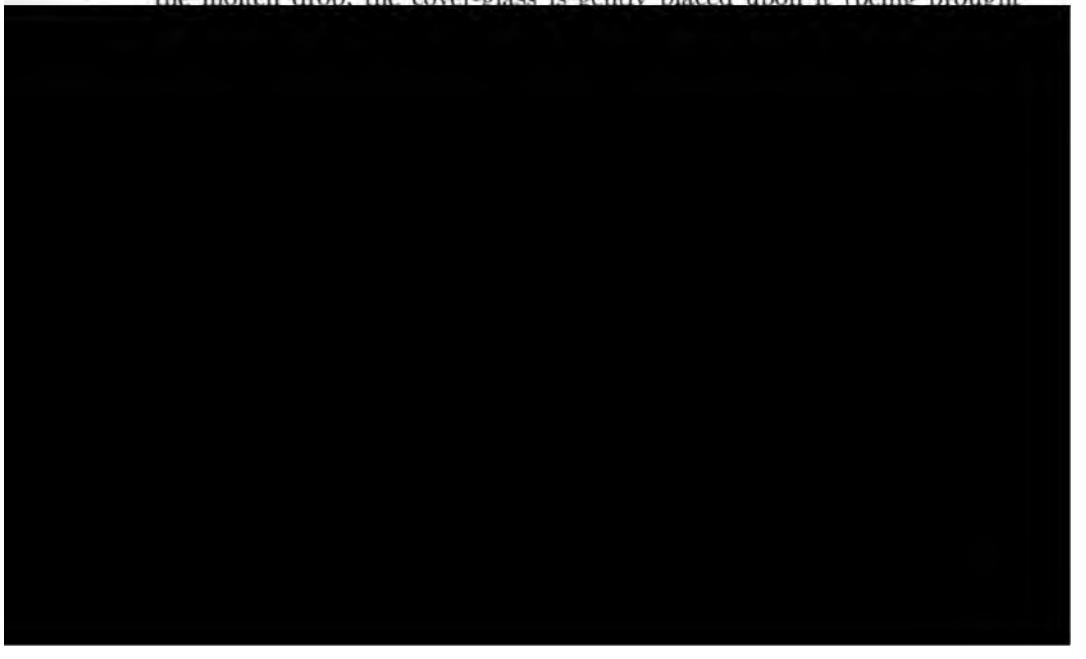
Experience will soon show how far this rubbing out should be carried for the best effects. Gentle pressure should be applied, care being taken not to break the cover-glass, especially if the sample contain anything of a gritty nature. The rubbing should be continued till the coarser par-

ticles and overlying masses are separated and distributed uniformly, but if too long persisted in, the forms of the tissues, starch grains, and other characteristic portions will be partially destroyed and of too fragmentary a nature to be readily recognizable.

Canada Balsam is the best mountant for the examination of starches under polarized light. In this medium, under ordinary illumination, the starches are not plainly visible, since the refractive index of the balsam is so near that of the starch grains themselves. With the crossed nicols, however, the starch grains stand out very clearly and distinctly in a dark background.

Specimens to be mounted in Canada balsam must be free from moisture. Dehydration is often resorted to by soaking the specimens in alcohol. Canada balsam in solution is prepared by dissolving the balsam broken into small pieces or powdered in a mortar in an equal volume of xylol, filtering and evaporating to sirupy consistency at room temperature.

*Glycerin Jelly.**—This is the best permanent mountant for powdered food substances and is prepared as follows: 1 part by weight of the finest French gelatin is soaked two hours in 6 parts of distilled water, after which 7 parts by weight of C. P. glycerin are added, and to each 100 parts of the mixture add 1 part of concentrated carbolic acid. Heat the mixture while stirring till flocculency disappears and filter through asbestos while warm, the asbestos being previously washed and put into the funnel while wet. The jelly is solid at ordinary temperatures, and must be warmed to melt. A small bit of this jelly is removed from the mass by a knife-blade and placed on the cover-glass, which is held over a gas flame till the jelly is melted. The powdered specimen being then shaken into the molten drop, the cover-glass is gently placed upon it (being brought



is but a mechanical mixture of various tissues, and that no two portions will show exactly the same composition.

Characteristic Features of Vegetable Foods under the Microscope.—The structural features of a powdered spice, examined microscopically, will be found to differ considerably in appearance from those of a thin, carefully mounted section of the same spice. Instead of the beautiful arrangement of cells and cell contents with the perfect order of various parts as seen in the mounted section, one finds in the powdered sample under the microscope what often appears to be a most confusing mass of fragments of various tissues. For this reason the most striking characteristics seem to vary with different observers, and it is a well-known fact that microscopists differ widely as to conceptions of size, shape, and ordinary appearance, even in the case of certain of the well-known starch grains. It is on this account that, irrespective of the description of others, the analyst should familiarize himself with the microscopical appearance of the foods with which he is dealing, as well as of their adulterants, forming his own standards as to what constitute the recognizable features, from specimens prepared by himself.

In the large variety of ground berries, buds, tubers, barks, etc., from which the spices and condiments are prepared, as well as in the grains, legumes, shells, fruit stones, and other materials forming the most familiar adulterants, the kinds of plant tissues and cell contents which, under the microscope, serve as distinguishing marks or guides for identification are comparatively few in number.

The most common of these varieties of cell tissue and of cell contents to be met with by the food microscopist in his work are briefly the following:

Parenchyma.—This is most abundant and widely distributed, forming as it does the thin-walled, cellular tissue of nearly all vegetable food substances. The walls of parenchyma cells are, as a rule, colorless and transparent. The forms of the cells are varied and are often sufficiently characteristic in themselves to identify the substance under examination.

Sclerenchyma, or stone cells, are the thick-walled woody cells forming the hard part of nut shells, fruit stones, and seed coverings, occurring also in some fruits and barks. These cells are more often colored and of various shapes but almost always irregular, sometimes elongated, as in cocoanut shells and olive stones occasionally nearly rectangular, as in pepper shells, and sometimes polygonal or nearly circular.

In appearance the sclerenchyma cell commonly has a more or less

deep, central or axial rift, from which small fissures extend through the thick walls, somewhat suggestive of the iris. See Fig. 27.

Variously shaped sclerenchyma cells are found in allspice, cassia,

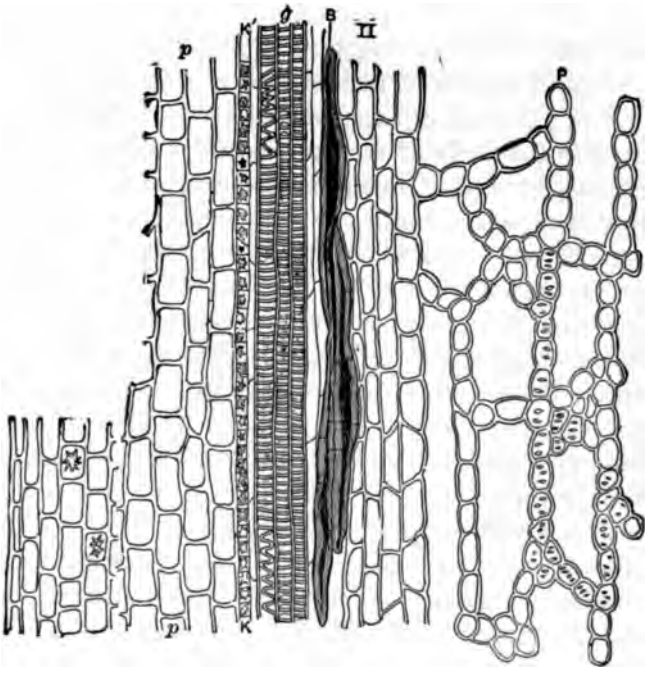


FIG. 26.—Typical Forms of Various Cell Tissues. Longitudinal section through a clove, showing: *Pp*, two forms of parenchyma; *B*, bast fibers; *g*, vascular and sieve tissue; *KK'*, cells with calcium oxalate crystals. (After Vogl.)

pepper, clove stems, nut shells, etc. Stone cells are optically active to polarized light, and between crossed nicols are very conspicuous by their bright appearance.

occurring in the bark (or phloem), and the wood fibers, in the woody portion (or xylem) of flowering plants. Bast fibers are common in cassia and ground bark, and wood fibers are met with in ginger and turmeric and such adulterants as sawdust.

Corky Tissue, or Suberin, constitutes the thin-walled, spongy cells forming the protective, outer dead layers of the bark. This is found in cassia, and in the barks used as adulterants. Suberin is tested for by potassium hydroxide (p. 81).

Spiral and Reticulated Ducts, and Sieve Tissues of various forms (suggested by the names), occur commonly interspersed among the parenchyma and vascular tissue. Notable examples of them are the spiral ducts found in chicory.

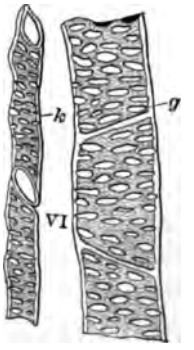


FIG. 28.—Reticulated Ducts of Chicory. (After Vogl.)

Starch wherever it occurs furnishes the most characteristic feature of the cell contents, and, as a rule, will at once indicate under the microscope, by the shape and grouping of its granules, the particular substance of which it forms a part. It is very abundantly distributed throughout the vegetable kingdom and occurs in a wide variety of forms. It is particularly conspicuous when viewed by polarized light. Between crossed nicols such starches as corn, potato, and arrowroot show out brightly from a dark background with dark crosses, the bars of which intersect at the hylum of each granule. When a selenite plate is introduced above the polarizer, a beautiful play of colors is seen with various starches, a phenomenon which Blyth applies as a means of identification and classification, but which more modern microscopists regard as of minor importance to distinguishing the various starches morphologically. Starch is found naturally in the cereals, legumes, and many vegetables, in cassia, allspice, nutmeg, pepper, ginger, cocoa, and turmeric. The cereal and leguminous starches from their inertness and cheapness constitute the most common adulterants of the spices and of powdered foods in general. Starch grains are found in the cells of the parenchyma and in other cellular tissues. Iodine is the special reagent (p. 79).

Gums and Resins occur in characteristic forms among the cell contents of some of the spices. As an example, the portwine-colored lumps of gum in allspice furnish one of the most ready means of recognizing that spice microscopically. Resin is tested for microchemically with alkanna tincture (p. 80).

Aleurone, or Protein Grains, occur in some of the spices, but are not especially characteristic. They somewhat resemble small starch grains. Most varieties of protein grains are soluble in water, but some are insoluble. The soluble varieties, which are not apparent in water-mounted specimens, must be examined in absolute alcohol, glycerin, or oil. In leguminous seeds aleurone occurs closely intermingled with starch in the same cells, while in the cereals it occupies the whole cell.

Protein grains are tested for under the microscope by iodine in potassium iodide, which turns them brown or yellowish brown, and by Millon's reagent, which colors them brick red.

Plant Crystals are not uncommon in the class of substances which the food analyst examines. The most common of these are the piperin crystals found in pepper. Needle-shaped calcium oxalate crystals, termed *raphides,* commonly grouped in bundles, are found occasionally in powdered cassia, and also in the onion and the beet.

Crystals of calcium carbonate are sometimes met with also, as, for example, in hops. Calcium oxalate crystals are insoluble in acetic acid, while being readily soluble in dilute hydrochloric. Calcium carbonate crystals are soluble with effervescence in both acids. The acid reagents are directly applied to the sample in water-mount under the cover-glass, and the reaction observed through the microscope.

Fat Globules are of common occurrence in many foods and appear of various sizes, sometimes large and conspicuous, and again almost lost sight of because of their minuteness. They are sometimes colorless, as in mace, and sometimes deeply tinted, as in cayenne. Alkanna tincture is used as a reagent for fat (p. 80).

Other Cell Contents of less importance, but which may be identified by the microscope with reagents, are tannic acid (tested for by chloriodide

Analytical reagents are applied to the water-mounted sample by means of a glass rod or pipette, with which a drop of the reagent is deposited on the sample upon the slide, having previously removed the cover, which is afterwards replaced. Or, without removing the cover-glass, a drop of the reagent is placed in contact with one side of it on the slide. Along the opposite side of the cover is then placed a piece of filter-paper. The latter withdraws by capillary attraction a portion of the water from under the cover-glass, and this is replaced by the reagent, which thus intermingles with the particles of the substance.

Following is a brief list of the commoner microchemical reagents, together with their method of preparation and chief uses. For fuller details in this branch of the subject the reader is referred to Poulsen's Botanical Microchemistry, translated by Trelease, and Zimmerman's Botanical Microtechnique.

A. Analytical Reagents.—*Iodine in Potassium Iodide.*—Two grams of crystallized potassium iodide are first dissolved in 100 cc. of distilled water and the solution is saturated with iodine.

This reagent is indispensable for the identification of starch, especially when the latter is present in minute quantities. Starch granules when moistened with water are turned blue by iodine, the reaction being exceedingly delicate under the microscope, even when the starch granules are very minute and insignificant without the reagent.

Iodine in connection with sulphuric acid is also useful in distinguishing pure cellulose from its various modifications, such as lignin and suberin. For this purpose the water-mounted sample is first permeated with concentrated sulphuric acid, after which the iodine reagent is applied, with the result that all pure cellulose is turned a deep-blue color, while the modified forms of cellulose are colored yellow or brown. The cellulose is first converted by the sulphuric acid into a carbohydrate isomeric with starch, known as amyloid.

Protein grains are colored brown or yellow brown by the action of iodine.

Chloriodide of Zinc.—Pure zinc is dissolved in concentrated hydrochloric acid to saturation, and an excess of zinc added. The solution is then evaporated to about the consistency of concentrated sulphuric acid, after which it is first saturated with potassium iodide, and finally with iodine.

This reagent may be used instead of sulphuric acid and iodine for the

detection of cellulose, since the zinc chloride converts the cellulose into amyloid, which the reagent colors blue.

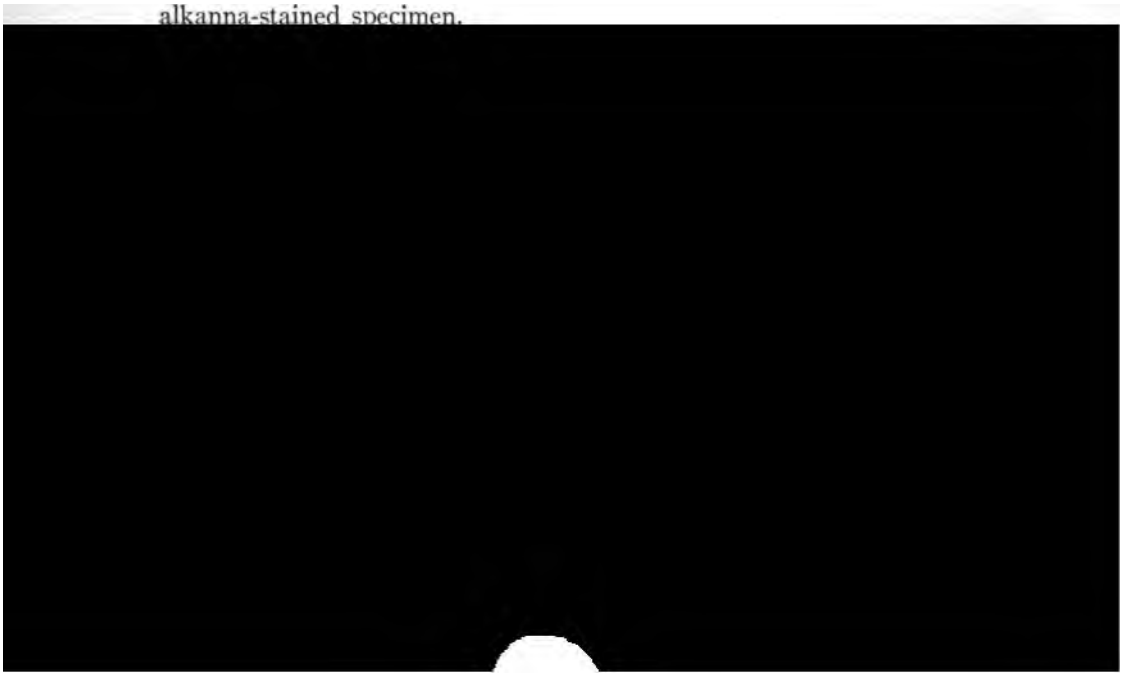
Chloriodide of zinc is useful for detecting tannic acid in cell contents. For this purpose the above reagent is much diluted by the addition of a 20% solution of potassium iodide. In this diluted form, when applied to the sample, a reddish or violet coloration is imparted to cell contents having tannin.

Phenol-hydrochloric Acid is prepared by saturating concentrated hydrochloric acid with the purest crystallized carbolic acid. Wood fiber, or lignin, when treated with a drop of this reagent under the cover-glass, and exposed for half a minute to the direct sunlight, will be colored an intense green, which quickly fades.

Indol.—Several crystals of indol are freshly dissolved in warm water. Lignified cell walls assume a deep-red color, when the specimen to be examined is treated first with a drop of the indol reagent, and afterwards washed with dilute sulphuric acid, 1:4.

Millon's Reagent.—This is prepared by dissolving metallic mercury in its weight of concentrated nitric acid, and diluting with an equal volume of water. This reagent, which should be freshly prepared, is of use in testing for protein compounds, which turn brick red when treated with it, especially on gently warming the slide.

Tincture of Alkanna.—A 70 or 80% alcoholic extract of alkanna root, when kept in contact with resins, fixed oils, fats, or essential oils for a short time, stains these cell contents a lively red. The staining is hastened by the aid of heat. Essential oils and resins are soluble in strong alcohol, while fixed oils and fats are insoluble, hence the distinction between these classes of cell contents may be made by the application of alcohol to the alkanna-stained specimen.



Chloral Hydrate.—A 60% solution.

Ammonia.—Concentrated, or 28% ammonia is commonly used.

Potassium Hydroxide, used in various degrees of concentration, often in dilute solution, say 5%. This reagent causes swelling of the cell wall, and acts as a solvent of intercellular substances as well as of protein. It bleaches most of the coloring matters, destroys the starch, and forms soluble soaps with the fats. Potassium hydroxide is also used in testing for suberin, which is extracted from corky tissue on boiling with the reagent, and appears as yellow drops.

Schultze's Macerating Reagent (concentrated nitric acid and chlorate of potassium) is best used by placing the powder or bit of tissue to be treated in a test-tube with an equal volume of potassium chlorate crystals, adding about 2 cc. of concentrated nitric acid, and warming the tube till bubbles are evolved freely, or until the necessary separation of cells is effected. The sample is then removed and washed with water.

By this treatment, bast and wood fibers as well as stone cells are readily separated from other tissues.

Cuprammonia (Schweitzer's Reagent).—This is prepared by adding slowly a solution of copper sulphate to an aqueous solution of sodium hydroxide, forming a precipitate of cupric hydroxide, which is separated by filtration, washed, and dissolved in concentrated ammonia. It should be freshly prepared, and is never fit for use unless it is capable of immediately dissolving cotton. Indeed its chief use is as a test for cellulose, which it readily dissolves. In observing this reaction under the microscope, the powdered specimen under the cover-glass should be only slightly damp before a drop of the fresh reagent is applied. The cell walls are seen to swell up and gradually become more and more indistinct, till they finally disappear.

Cuprammonia is also used as a test for pectose, which occurs in many cell walls, often intermixed with cellulose. When treated with this reagent, cellular tissue containing pectose is acted upon in such a manner that a delicate framework of cupric pectate is sometimes left behind, after the dissolution of the cellulose with which it is mingled.*

Photomicrography.—The photomicrograph serves as a simple means of keeping permanent records of unusual forms of adulteration met with during routine examination. Besides this, the photomicrograph has at times proved its usefulness as a means of evidence in court, showing as it does with faithfulness the presence of a contested adulterant. It is true

* Poulsen, Botanical Micro-chemistry, p. 15.

that from an artistic standpoint the photomicrograph of a powdered sample is often disappointing, due to the fact that ordinarily much of the field is out of focus, unless a very simple homogeneous subject is photographed, as, for instance, starch. As compared with the carefully prepared drawing of a section, which is usually idealized, the photomicrograph is of course the more truthful representation.

SUMMARY OF MICROCHEMICAL REACTIONS FOR IDENTIFYING CELLULAR TISSUE AND CELL CONTENTS. BASED ON BEHRENS'.*

	Iodine in Potassium Iodide.	Chlor-iodide of Zinc.	Iodine and Sulphuric Acid.	Cupram-monia.	Potassium Hydroxide.	Concentrated Sulphuric Acid.	Schultze's Mixture.
Cellulose, cell substance.	Yellow to brownish	Violet	Blue	Dissolves	Swells up	Dissolves	Dissolves
Lignin, wood substance.	Yellow	Yellow	Yellow to brownish	Insoluble	Dissolves	Dissolves	Dissolves easily
Middle lamella, intercellular substance.	Yellow	Yellow	Yellow	Insoluble	.....	.....	Dissolves easily
Suberin, cork substance.	Yellow or brownish	Yellow or brown	Brown	Insoluble	Insoluble in cold. By boiling it comes out in drops	Insoluble	Gives ceric acid reaction†
Starch.	Blue	.....	.....	.....	Dissolves	.....	.....
Protein.	Brown yellow	.....	.....	.....	Dissolves	.....	.....
Gums and resins.	.....	.....	.....	.....	.....	.....	.....
Fat.	.....	.....	.....	.....	Saponifies	.....	.....
Essential oils.	.....	.....	.....	.....	.....	.....	.....
Tannin.	.....	Reddish to violet	.....	.....	.....	.....	.....
Calcium oxalate crystals	.....	.....	.....	.....	.....	.....	.....
Calcium carbonate "	.....	.....	.....	.....	.....	.....	.....

	Phenol-hydro-chloric Acid.	Indol.	Ferric Acetate or Sulphate.	Alkanna Tincture.	Hydro-chloric Acid.	Acetic Acid.	Millon's Reagent.
Cellulose, cell substance.	Uncolored	Uncolored	.....	.....	.....	.....	.....
Lignin, wood substance.	Green	Cherry red	.....	.....	.....	.....	.....
Middle lamella, intercellular substance.	Green	Cherry red	.....	.....	.....	.....	.....

he does unconsciously, so that he does not realize how far from flat the field actually is till he undertakes to photograph it, when, as a rule, only a small portion is in good focus. It is therefore impossible in one photograph to show successively many varied forms of tissue or cell contents in the powder, but separate photographs should be made as far as possible with only a little in each. Thus, for example, with a composite subject like powdered cassia bark, it would be very difficult to show starch, stone cells, and bast fibers in one field, all in equally good focus, and, for the best results only, one, or at most two, such varied groups of elements should be shown in one picture.

Appurtenances and Methods of Procedure.—The temporary method of water-mounting employed by the analyst in routine examination presents many difficulties from a photographic point of view. The vibrating motion of the particles is very annoying, and some skill is required in using just the right amount of water, in avoiding air-bubbles, in waiting the requisite amount of time before exposing the plate for the vibratory motion to cease, and, on the other hand, avoiding too long delay, which would result in the evaporation of the water, and the consequent breaking up of the field. In the writer's experience, however, in spite of these difficulties, the water-mounting gives decidedly the clearest results, and, with patience on the part of the operator, it is in many ways the most desirable method of mounting for photographic purposes. It is in fact the method employed in making most of the photomicrographs of powdered specimens that appear in the plates at the end of this volume, though a few were mounted in glycerin jelly, and the starches for the polarized-light pictures in Canada balsam. The sections of tissues shown in the plates were mounted some in glycerin and others in glycerin jelly.

Experience has shown that two degrees of magnification well calculated to bring out the chief characteristics of the spices and their adulterants in a photomicrograph are 125 and 250 diameters. The starches, which are the most common of any one class of adulterants, vary very widely in the size of their granules. With these the larger magnification of 250 has been found satisfactory. while the general appearance of the composite ground-spice itself under the microscope, as well as that of such adulterants as ground bark, sawdust, chicory, pea hulls, and the like, is best shown with the lower power of 125.*

* The degrees of magnification adopted in the originals of most of the photomicrographs illustrated in the accompanying plates are accordingly 125 and 250, but in the process of lithographing, the photographs were slightly reduced, so that the actual scales in the reproduction are 110 and 220 respectively.

The object, mounted in the manner above described, is best examined when held in a mechanical stage, furnished with micrometer adjustments, in such a manner that a typical field may be selected and held in place long enough to photograph.

The Camera.—This need not of necessity be complicated, but may consist simply of a horizontal wooden base on which the microscope



The base is a solid iron plate upon which the microscope rests (any microscope may be used with this camera), and above which the camera bellows is supported on two solid steel rods. The bellows is supported at either end on wooden frames.

The ground glass is provided with a central transparent area, formed by cementing a cover-glass upon the ground glass, and permits the accurate focusing of the most delicate detail by means of a hand magnifying-glass. The vertical rods supporting the bellows are attached to metal arms, immovably fixed to a horizontal axis, thus permitting the camera to be tilted



FIG. 30.—Photomicrographic Camera in Horizontal Position

to any angle from vertical to horizontal. It is fixed at the desired angle by means of heavy hand-clamps.

In use the camera is placed in a vertical position and the microscope adjusted on the base so that its tube will coincide with the opening in the front of the camera. The connection between microscope and camera is made light-tight by means of a double chamber, which permits considerable vertical motion of the tube of the microscope without readjustment. A jointed telescoping rod is attached to the upper end of the camera to act as a support, giving perfect steadiness when in a horizontal position, and folding down parallel with the bellows so as to be out of the way when in any other position.

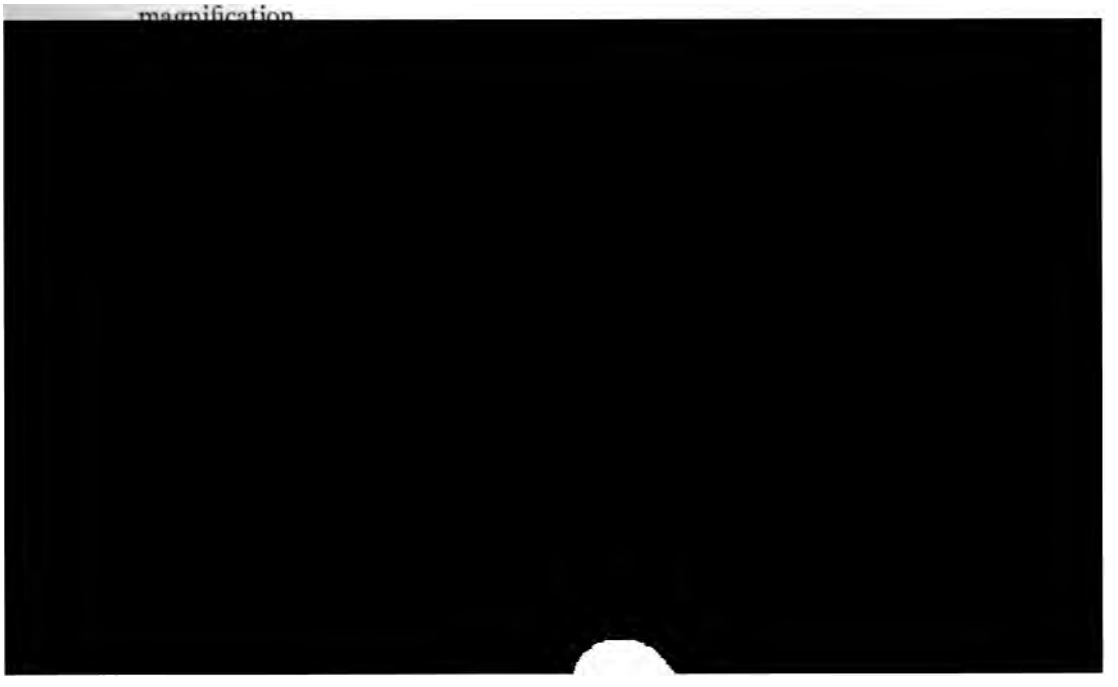
Amplification.—The vertical rods are graduated in inches for determining the amount of amplification, and to show when the ground glass is at right angles to the optical axis. The following simple rule for determining the amount of amplification will give sufficiently accurate results. When photographing without the eyepiece, divide the distance of the ground glass from the stage of the microscope in inches, by the focal length in inches of the objective used. When photographing with the eyepiece, proceed as above and multiply the result by the quotient obtained by dividing 10 by the focus in inches of the eyepiece used.

Adjustment and Manipulation.—The microscope can be placed in any position desired, and the camera adjusted to it. The bellows can then be raised and the microscope used as though no camera were present. When an object is to be photographed, the bellows may be slid into position without in any way disturbing the arrangement of light or object, the final focusing on the ground glass being effected quickly by means of the fine adjustment-screw of the microscope. The exposure having been made, observation through the microscope may be continued without interruption by simply raising the bellows again.

When a water-mounted specimen is to be photographed, the camera and microscope tube should be vertical instead of inclined as shown in the cut.

The camera is best kept in a dark room where the exposures are to be made, the source of light being a 16- or 32-candle-power electric lamp, preferably provided with a ground-glass bulb. The light from this lamp is first carefully centered by moving the reflector of the microscope.

In making pictures, for instance, of the magnification of 250 diameters, the objective, having an equivalent focus of $\frac{1}{6}$ inch, may be used in combination with the one-inch ocular, with the ordinary tube length of microscope. For a lower power, such as 125 diameters, the same objective is employed, but the eyepiece is left out, it being found necessary in this case to remove the upper tube of the microscope, which ordinarily carries the eyepiece, as otherwise the size of the field to be photographed would be restricted. In each case a diaphragm is used in the microscope stage, having an opening of about the same size as that of the front lens of the objective. By means of a stage micrometer scale, the proper position of the camera back is previously determined to give the required magnification.



CLARK, C. H. Practical Methods in Microscopy, 1896.
DAMMAR, O. Illustrirtes Lexicon der Verfälschungen und Vereinigungen der Nahrungs- und Genussmittel. Leipzig, 1886.
DETMER, W. Das pflanzenphysiologische Praktikum. Jena, 1885.
DIETSCH, O. Die wichtigsten Nahrungsmittel und Getränke, deren Verunreinigungen und Verfälschungen. Zurich, 1884.
GAGE, S. H. The Microscope and Microscopical Methods. Ithaca, 1894.
GREENISH, H. G. The Microscopical Examination of Foods and Drugs. Philadelphia, 1903.
HAUSHOFER, K. Mikroskopische Reaktionen. Braunschweig, 1885.
HEGLER. Histochemische Untersuchungen verholzter Zellmembranen. Flora, 1890, page 31.
HOFFMEISTER, T. Die Rohfaser und einige Formen der Cellulose. Landwirtschaftl. Jahrbücher, 1888, page 239.
KOCH, L. Mikrotechnische Mittheilungen. Pringsheim's Jahrbücher, Bd. XXIV, page 1, 1892.
KRAEMER, H. Botany and Pharmacognosy. Philadelphia, 1903.
KRAUS, G. Zur Kentniss der Chlorophyllfarbstoffe. Stuttgart, 1872.
LANGE, G. Zur Kentniss des Lignins. Zeits. für physiologische Chemie. Bd. XIV, page 15.
LEACH, A. E. Microscopical Examination of Foods for Adulteration. An. Rep. Mass. State Board of Health, 1900, p. 679.
LEE, A. B. The Microtomist's Vade Mecum. 1893.
MACÉ, E. Les Substances Alimentaire Etudiés au Microscope. Paris, 1891.
MOELLER, J. Mikroskopie der Nahrungs- und Genussmittel aus dem Pflanzenreiche. Berlin, 1886.
—— Pharmacognostischer Atlas. Berlin, 1892.
MOLISCH. Grundriss einer Histochemie der pflanzlichen Genussmittel. Jena, 1891.
NEUHAUSS, R. Lehrbuch der Mikrophotographie. Braunschweig, 1890.
POULSEN, V. A. Botanical Microchemistry, translated by Trelease. Boston, 1886.
PRINGLE, A. Practical Photomicrography. New York, 1890.
SCHIMPER, A. F. W. Mikroskopischen Untersuchungen der Nahrungs- und Genussmittel. Jena, 1886.
STRASSBURGER, E. Manual of Vegetable Histology, translated by Hervey. 1887.
THOMAS and DUDLEY. A Laboratory Manual of Plant Histology.
TSCHIRCH, A., und OESTERLE, O. Anatomischer Atlas der Pharmakognosie und Nahrungsmittelkunde. Leipzig, 1900.
VOGL, A. E. Die wichtigsten vegetabilischen Nahrungs- und Genussmittel. Berlin, 1899.
WORMLEY, T. G. The Microchemistry of Poisons. Philadelphia, 1885.
ZIMMERMAN, A. Botanical Microtechnique. New York, 1893.
—— Die Morphologie und Physiologie der Pflanzenzelle. Breslau, 1887.
—— Beiträge zur Morphologie und Physiologie der Pflanzenzelle. Tübingen, 1890.

CHAPTER VI.

MILK.

Nature and Composition.—Milk is the secretion of the mammary glands of female mammals for the nourishment of their young. Containing as it does all the requisites for a complete food, i.e., sugar, fat, proteids, and mineral ingredients, combined in appropriate proportion, there is ample reason why it occupies so high a place in the scale of human foods. It is a yellowish-white opaque fluid, denser than water, containing in complete solution the sugar, soluble albumin, and mineral content, and, in less complete solution, the casein, while the fat-globules are held in suspension in the serum, forming an emulsion.

The specific gravity of pure milk ranges from 1.027 to 1.035.

Milk from various animals has the same general physical properties and the same ingredients, differing, however, in percentage composition. Of all the varieties, the milk of the cow is by far the most important from its universal use, and, unless otherwise qualified, the term milk wherever it occurs in this volume will be understood to mean cow's milk.

Acidity.—When perfectly fresh, milk of carnivorous mammals is, as a rule, acid in reaction, while human milk and that of the herbivora is



Color.—The yellow color of milk is imparted to it by the fat globules, and varies greatly in milk from different breeds of cattle, as well as in milk from the same cow at different seasons, being, as a rule, paler during the winter or stall-fed months, and having its greatest intensity soon after the cow is put out to pasture.

Milk Sugar, the carbohydrate of milk, is normally present in amounts varying from 3 to 5 per cent. For the properties of milk sugar see page 472.

The Proteids of Milk.—*Casein* constitutes nearly 80% of the entire proteids of milk, being present in an average sample to the extent of about 3%. It exists in combination with calcium phosphate, and probably does not form a perfect solution in the milk, but is rather diffused therein in a somewhat colloidal form, being so finely divided, however, as to be incapable of separation by filtration while the milk is fresh.

Pure casein is a white, odorless, and tasteless solid, sparingly soluble in water, and insoluble in ether and alcohol. It is readily soluble in dilute alkalies. Strong acids also dissolve it, but its character is changed. From alkaline solution it is precipitated without change by neutralizing with acid. Its solutions are lævo-rotary.

Lact-albumin is the soluble albumin of milk, existing therein to the extent of about 0.6% and forming about 15% or more of the milk proteids. It much resembles the albumin of eggs, being coagulated at 70° to 75° C. It is readily soluble in water. Its specific rotary power according to Béchamp is $[\alpha]_D = -67.5$.

Lactoglobulin has been discovered by Emmerling as a constituent in milk, but exists in traces only. According to Babcock, it may be separated from milk whey by carefully neutralizing with sodium hydroxide, and afterwards saturating with magnesium sulphate. It much resembles the globulin of blood serum, being coagulated at 67° to 76° C.

Fibrin.—Babcock has discovered in milk very minute traces of a substance analogous to the fibrin of blood. This substance, it is claimed, forms a part of the slime found in the separator-bowl of a centrifugal skimmer.

Other Nitrogenous Substances.—Besides the above normal constituents of milk. certain bodies may be formed by proteolytic action during fermentation, such, for example, as caseoses and peptones, formed for the most part by the decomposition of a part of the casein. Galactin is a gelatin-like body of the nature of peptone, occurring in traces in milk. Besides these, minute traces of amido-bodies, such as creatin and urea, are sometimes present, and also ammonia.

BLE SHOWING THE COMPOSITION OF MILK.*

Olein, Palmitin, Stearin, Myristin, Butin (trace)	Glycerides of insoluble and non-volatile acids.	3.3	Fat 3.6	Total solids 12.7
Butyrin, Caproin, Caprylin (trace), Caprinin (trace)	Glycerides of soluble and volatile acids.	0.3		
Casein 3.00; Albumin 0.60; Lactoglobulin, Galactin, Fibrin (trace) 0.20	containing nitrogen.	3.8	Solids not fat 9.1	
Milk sugar		4.5		
Citric acid		0.1		
Potassium oxide 0.175; Sodium oxide 0.070; Calcium oxide 0.140; Magnesium oxide 0.017; Iron oxide 0.001; Sulphur trioxide 0.027; Phosphoric pentoxide 0.170; Chlorine 0.100	Ash	0.7		
Water		87.3		
		100.0		

* According to S. M Babcock.

Milk Fat.—Fat forms the most variable constituent of milk, being found in proportions ranging from 2.5 to 7 per cent. For the chemical composition and characteristics of milk fats see Butter (p. 431).

The fat globules are held in suspension in the milk and have long been thought to be surrounded each by a thin nitrogenous membrane, known as *Storch's mucoid proteid*, which becomes broken on churning. This theory, while rendered probable by many of the phenomena connected with the dairy, is by no means universally held at present.

Citric Acid has been found to exist in milk, probably in combination with certain of the mineral constituents, being present to the extent of about 0.1%.

The table on page 90 arranged by Babcock shows quite clearly the percentage composition of an average cow's milk.

For comparison of milk from different animals the following table* is inserted, showing in most cases minimum, maximum, and mean determinations from a large number of actual analyses:

	No. of Analyses.	Specific Gravity.	Water.	Casein.	Albumin.	Total Proteids.	Fat.	Milk Sugar.	Ash.
Cow's milk	800								
Minimum		1.0264	80.32	1.79	0.25	2.07	1.67	2.11	0.35
Maximum		1.0370	90.32	6.29	1.44	6.40	6.47	6.12	1.21
Mean		1.0315	87.27	3.02	0.53	3.55	3.64	4.88	0.71
Human milk	200								
Minimum		1.027	81.09	0.18	0.32	0.69	1.43	3.88	0.12
Maximum		1.032	91.40	1.96	2.36	4.70	6.83	8.34	1.90
Mean			87.41	1.03	1.26	2.29	3.78	6.21	0.31
Goat's milk	200								
Minimum		1.0280	82.02	2.44	0.78	.....	3.10	3.26	0.39
Maximum		1.0360	90.16	3.94	2.01	.....	7.55	5.77	1.06
Mean		1.0305	85.71	3.20	1.09	4.29	4.78	4.46	0.76
Ewe's milk	32								
Minimum		1.0298	74.47	3.59	0.83	.....	2.81	2.76	0.13
Maximum		1.0385	87.02	5.69	1.77	.....	9.80	7.95	1.72
Mean		1.0341	80.82	4.97	1.55	6.52	6.86	4.91	0.89
Mare's milk	47								
Mean		1.0347	90.78	1.24	0.75	1.99	1.21	5.67	0.35
Ass's milk	5								
Mean		1.036	89.64	0.67	1.55	2.22	1.64	5.99	0.51

Composition of the Ash of Milk.—The ash of milk does not truly represent the mineral content, since, in the process of incineration, the character of some of the constituents is altered by oxidation and otherwise.

Expressed in parts per 100, the ash of the typical milk sample whose full analysis is given on page 90 would be about as follows:

* Compiled from König's Chemie der mens. Nahr. u. Genuss.

Potassium oxide	25.02
Sodium "	10.01
Calcium "	20.01
Magnesium "	2.42
Iron "	0.13
Sulphur trioxide	3.84
Phosphoric pentoxide	24.29
Chlorine	14.28
	100.00

Soldner regards the following as more nearly representing the proportion in which the mineral salts exist in milk:

	Per Cent.
Sodium chloride, NaCL	10.62
Potassium chloride, KCL	9.16
Mono-potassium phosphate, KH_2PO_4	12.77
Di-potassium phosphate, K_2HPO_4	9.22
Potassium citrate, $K_3(C_6H_5O_7)_2$	5.47
Di-magnesium phosphate, $MgHPO_4$	3.71
Magnesium citrate, $Mg_3(C_6H_5O_7)_2$	4.05
Di-calcium phosphate, $CaHPO_4$	7.42
Tri-calcium phosphate, $Ca_3(PO_4)_2$	8.90
Calcium citrate, $Ca_3(C_6H_5O_7)_2$	23.55
Lime, combined with proteids	5.13
	100.00

Fore Milk and Strippings.—Unless a portion drawn from the well-



The per cent of albuminoids, sugar, and ash is nearly the same in both fore milk and strippings.

Colostrum.—The milk given by cows and other mammals for two or three days after the birth of young is termed colostrum, and differs materially in composition from normal milk. It is yellow in color, of an oily consistency, and has a pungent taste. It acts as a purge upon the young. Examined under the microscope, it is found to contain large circular cells larger than fat globules and somewhat similar to blood corpuscles. It is very high in albumin, which seems to be similar to blood albumin. The following analyses were made by Engling, showing the composition of colostrum from a cow eight years old:

Time after Calving.	Specific Gravity.	Fat.	Casein.	Albumin.	Sugar.	Ash.	Total Solids.
Immediately.....	1.068	3.54	2.65	16.56	3.00	1.18	26.93
After 10 hours.....	1.046	4.66	4.28	9.32	1.42	1.55	21.23
" 24 "	1.043	4.75	4.50	6.25	2.85	1.02	19.37
" 48 "	1.042	4.21	3.25	2.31	3.46	0.96	14.19
" 72 "	1.035	4.08	3.33	1.03	4.10	0.82	13.36

The average of twenty-two analyses of colostrum from different cows by Engling showed total solids 28.31, fat 3.37, casein 4.83, albumin 15.85, sugar 2.48, ash 1.78.

Frozen Milk.—Since it is the water in milk that freezes, it follows that in partially frozen milk the unfrozen portion of the milk, or that part which remains still liquid, becomes concentrated by the process of freezing. This is borne out by the following figures of Richmond:*

	Frozen Portion, Per Cent.	Unfrozen Portion, Per Cent.
Water.....	96.23	85.62
Fat.....	1.23	4.73
Sugar.....	1.42	4.95
Proteids.....	.91	3.90
Ash.....	.21	.80
Specific gravity.....	1.0090	1.0345

Fermentations of Milk.—These are due to the action of bacteria of various kinds, the most common being the lactic fermentation.

The Souring of Milk is caused by the action of a large number of species of acid-forming bacteria, chief among which is the *Bacillus acidi lactici*, which multiplies faster than other bacteria in raw milk under

* Analyst, XVIII. p. 53.

favorable conditions of temperature. Part of the milk sugar is acted on and transformed, first into dextrose and galactose, the latter sugar subsequently forming lactic acid, as follows:

(1) $\underset{\text{Lactose}}{C_{12}H_{22}O_{11}},H_2O = \underset{\text{Dextrose}}{C_6H_{12}O_6} + \underset{\text{Galactose}}{C_6H_{12}O_6}$

(2) $\underset{\text{Galactose}}{C_6H_{12}O_6} = 2\underset{\text{Lactic acid}}{C_3H_6O_3}$

More and more acid is formed until the casein can no longer be held up, curdling ensues, and the casein is precipitated. Finally, after a certain degree of acidity is reached, the ferment is killed and the action stops. Other acids than lactic are also undoubtedly produced, since a small part of the acid in sour milk is found to be volatile. According to Conn * the volatile acids are acetic and formic.

Abnormal Fermentation.—Through the agency of micro-organisms that may develop under certain conditions, various changes are produced in milk that to some extent alter its character. Thus *bitter milk* is sometimes produced as the result of some organism as yet but little understood.

Occasionally milk is found possessing a peculiarly thick and slimy consistency, whereby it may be drawn out in threads, by dipping a spoon into the milk and withdrawing it therefrom. This is termed *ropy* milk, and is more often met with in warm weather. It is undoubtedly produced as a result of bacterial action.

Enzyme-forming Bacteria are not uncommonly developed in milk, causing various proteolytic changes, whereby the casein is partially transformed into peptones, caseoses, etc.

Chromogenic Bacteria are the agencies that produce peculiar pigments in milk. Thus red milk is due to *Bacillus erythrogenes;* yellow milk to *Bacillus xynxanthus;* blue milk to *Bacillus cyanogenes.* The latter is



in the case of the milk sugar and the proteids, a determination of either one may be directly made (whichever is most convenient), the other being calculated by difference.

When foreign ingredients or adulterants are present in milk, special methods are employed to detect them.

Preparation of the Sample.—In procuring a sample for analysis, the greatest care is necessary to insure a homogeneous sample. By far the best method in every case, where possible, is to pour the milk back and forth from one vessel to another (i.e., pour from the original container into an empty vessel and back at least once). Where such procedure is impossible from the size of the container or for any other reason, a so-called "sampler" should be used, of which the Scovell sampling-tube is a convenient form. By means of this, a small sample for analysis representative of the whole is obtainable, which can afterwards be mixed by pouring.

This instrument consists of a brass or copper tube made in two parts which telescope accurately together as shown in Fig. 31, the lower part being closed at the bottom, but provided with three or more lateral slits. The sampler, drawn out to its full length, is carefully inserted in the tank containing the milk and lowered to the bottom, after which the upper part is pressed down over the lower so as to close the slits, and the tube is then lifted out of the tank, containing a fairly representative sample of the milk.

In all operations to which a milk sample is submitted during the process of analysis, it should invariably be poured into a clean empty vessel and back, whenever it has been at rest for an appreciable time, in order to insure a homogeneous mixture.

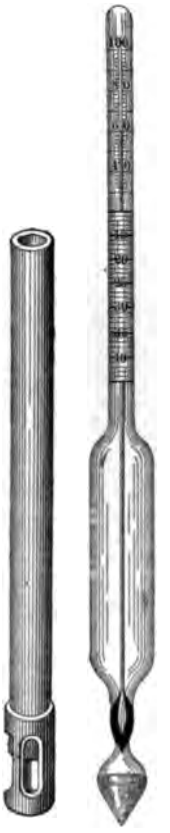


FIG. 31. FIG. 32.

FIG. 31.—The Scovell Milk-sampling Tube.

FIG. 32.—The Quevenne Lactometer.

Determination of Specific Gravity.—This is most readily obtained with the aid of a hydrometer, accurately graduated within the limits of the widest possible variation in the specific gravity of milk. Hydrometers for special use with milk are known as lactometers, and are graduated variously. One of the most convenient forms of this instrument is the Quevenne lactometer, graduated from 15° to 40°, corresponding to specific gravity 1.015 to 1.040. This

instrument, shown in Fig. 32, has a thermometer combined with it, the stem containing a double scale, on the lower part of which the specific gravity is read, while the temperature is read from the upper part.

Another form of instrument is termed the New York Board of Health lactometer, which is not graduated to read the specific gravity directly, but has an arbitrary scale divided into 120 equal parts, the zero being equal to the specific gravity of water, while 100 corresponds to a specific gravity of 1.029. To convert readings on the New York Board of Health scale to Quevenne degrees they must be multiplied by .29.

QUEVENNE LACTOMETER DEGREES CORRESPONDING TO NEW YORK BOARD OF HEALTH LACTOMETER DEGREES.

Board of Health Degrees.	Quevenne Scale.	Board of Health Degrees.	Quevenne Scale.	Board of Health Degrees.	Quevenne Scale.
60	17.4	81	23.5	101	29.3
61	17.7	82	23.8	102	29.6
62	18.0	83	24.1	103	29.9
63	18.3	84	24.4	104	30.2
64	18.6	85	24.6	105	30.5
65	18.8	86	24.9	106	30.7
66	19.1	87	25.2	107	31.0
67	19.4	88	25.5	108	31.3
68	19.7	89	25.8	109	31.6
69	20.0	90	26.1	110	31.9
70	20.3	91	26.4	111	32.2
71	20.6	92	26.7	112	32.5
72	20.9	93	27.0	113	32.8
73	21.2	94	27.3	114	33.1
74	21.5	95	27.6	115	33.4
75	21.7	96	27.8	116	33.6
76	22.0	97	28.1	117	33.9
77	22.3	98	28.4	118	34.2
78	22.6	99	28.7	119	34.5
79	22.9	100	29.0	120	34.8
80	23.2				

FOR CORRECTING THE SPECIFIC GRAVITY OF MILK ACCORDING TO TEMPERATURE (BY DR. PAUL VIETH).

Degrees of Lactometer.	Degrees of Thermometer (Fahrenheit).															
	45	46	47	48	49	50	51	52	53	54	55	56	57	58	59	60
20	19.0	19.0	19.1	19.1	19.2	19.2	19.3	19.4	19.4	19.5	19.6	19.7	19.8	19.9	19.9	—
21	19.9	20.0	20.0	20.1	20.2	20.2	20.3	20.3	20.4	20.5	20.6	20.7	20.8	20.9	20.9	—
22	20.9	21.0	21.0	21.1	21.2	21.2	21.3	21.3	21.4	21.5	21.6	21.7	21.8	21.9	21.9	—
23	21.9	22.0	22.0	22.1	22.2	22.2	22.3	22.3	22.4	22.5	22.6	22.7	22.8	22.8	22.9	—
24	22.9	22.9	23.0	23.1	23.2	23.2	23.3	23.3	23.4	23.5	23.6	23.6	23.7	23.8	23.9	—
25	23.8	23.9	24.0	24.0	24.1	24.1	24.2	24.3	24.4	24.5	24.6	24.6	24.7	24.8	24.9	—
26	24.8	24.9	24.9	25.0	25.1	25.1	25.2	25.2	25.3	25.4	25.5	25.6	25.7	25.8	25.9	—
27	25.8	25.9	25.9	26.0	26.1	26.1	26.2	26.2	26.3	26.4	26.5	26.6	26.7	26.8	26.9	—
28	26.7	26.8	26.8	26.9	27.0	27.0	27.1	27.2	27.3	27.4	27.5	27.6	27.7	27.8	27.9	—
29	27.7	27.8	27.8	27.9	28.0	28.0	28.1	28.2	28.3	28.4	28.5	28.6	28.7	28.8	28.9	—
30	28.6	28.7	28.7	28.8	28.9	29.0	29.1	29.1	29.2	29.3	29.4	29.6	29.7	29.8	29.9	—
31	29.5	29.6	29.6	29.7	29.8	29.9	30.0	30.1	30.2	30.3	30.4	30.5	30.6	30.8	30.9	—
32	30.4	30.5	30.5	30.6	30.7	30.9	31.0	31.1	31.2	31.3	31.4	31.5	31.6	31.7	31.9	—
33	31.3	31.4	31.4	31.5	31.6	31.8	31.9	32.0	32.1	32.3	32.4	32.5	32.6	32.7	32.9	—
34	32.2	32.3	32.3	34.4	32.5	32.7	32.9	33.0	33.1	33.2	33.3	33.5	33.6	33.7	33.9	—
35	33.0	33.1	33.2	33.4	33.5	33.6	33.8	33.9	34.0	34.2	34.3	34.5	34.6	34.7	34.9	—

	61	62	63	64	65	66	67	68	69	70	71	72	73	74	75
20	20.1	20.2	20.2	20.3	20.4	20.5	20.6	20.7	20.9	21.0	21.1	21.2	21.3	21.5	21.6
21	21.1	21.2	21.3	21.4	21.5	21.6	21.7	21.8	22.0	22.1	22.2	22.3	22.4	22.5	22.6
22	22.1	22.2	22.3	22.4	22.5	22.6	22.7	22.8	23.0	23.1	23.2	23.3	23.4	23.5	23.7
23	23.1	23.2	23.3	23.4	23.5	23.6	23.7	23.8	24.0	24.1	24.2	24.3	24.4	24.6	24.7
24	24.1	24.2	24.3	24.4	24.5	24.6	24.7	24.9	25.0	25.1	25.2	25.3	25.5	25.6	25.7
25	25.1	25.2	25.3	25.4	25.5	25.6	25.7	25.9	26.0	26.1	26.2	26.4	26.5	26.6	26.8
26	26.1	26.2	26.3	26.5	26.6	26.7	26.8	27.0	27.1	27.2	27.3	27.4	27.5	27.7	27.8
27	27.1	27.3	27.4	27.5	27.6	27.7	27.8	28.0	28.1	28.2	28.3	28.4	28.6	28.7	28.9
28	28.1	28.3	28.4	28.5	28.6	28.7	28.8	29.0	29.1	29.2	29.4	29.5	29.7	29.8	29.9
29	29.1	29.3	29.4	29.5	29.6	29.8	29.9	30.1	30.2	30.3	30.4	30.5	30.7	30.9	31.0
30	30.1	30.3	30.4	30.5	30.7	30.8	30.9	31.1	31.2	31.3	31.5	31.6	31.8	31.9	32.1
31	31.2	31.3	31.4	31.5	31.7	31.7	31.8	32.0	32.2	32.4	32.5	32.6	32.8	33.0	33.1
32	32.2	32.3	32.5	32.6	32.7	32.9	33.0	33.2	33.3	33.4	33.6	33.7	33.9	34.0	34.2
33	33.2	33.3	33.5	33.6	33.8	33.9	34.0	34.2	34.3	34.5	34.6	34.7	34.9	35.1	35.2
34	34.2	34.3	34.5	34.6	34.8	34.9	35.0	35.2	35.3	35.5	35.6	35.8	36.0	36.1	36.3
35	35.2	35.3	35.5	35.6	35.8	35.9	36.1	36.2	36.4	36.5	36.7	36.8	37.0	37.2	37.3

Determination of Total Solids.—For purposes of milk analysis, platinum dishes are by far the most desirable. These, if made for the purpose, should be of the shape shown in Fig. 33, measuring about 2¾ inches in

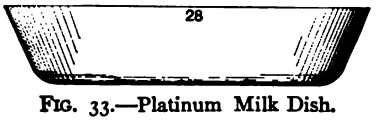


FIG. 33.—Platinum Milk Dish.

diameter at the top, and 2¼ inches in diameter at the bottom, having carefully rounded rather than square edges, and being ½ inch deep. The bottom is not perfectly flat, but slightly crowned outward. Such a dish will hold about 35 cc.

For purposes of economy it is best to have these dishes spun out with a thick bottom, but with thin sides, not so thin, however, as to be too readily bent.

If platinum dishes cannot be afforded, dishes of porcelain, glass, aluminum, nickel, or even tin may be used, but in all cases should be as thin as practicable.

About 5 cc. of the thoroughly mixed sample of milk are carefully transferred by means of a pipette to a tared dish on the scale-pan, and its weight accurately determined. The dish with its contents is then transferred to a water-bath, being placed over an opening preferably but little smaller than the diameter of the bottom of the dish, so that as large a surface as possible is in contact with the live steam of the bath. Here it is kept for at least two hours, after which the dish is wiped dry while still hot, transferred to a desiccator, cooled, and weighed.*

Determination of Ash.—The platinum dish containing the milk residue is next placed upon a suitable support above a Bunsen flame (a platinum triangle or a ring stand is convenient for this) and the residue is ignited at a dull-red heat to a perfectly white ash, after which it is cooled and weighed.

DETERMINATION OF FAT. — The Adams Method. — Without doubt the most accurate method of fat determination is the so-called Adams method. For this purpose a strip of fat-free filter-paper about 2½ inches wide and 22 inches long is rolled into a coil and held in place by a wire as shown in Fig. 34.

Schleicher and Schüll furnish fat-free strips especially for this work, but it is very easy to prepare the strips and extract them with the Soxhlet apparatus.

About 5 grams of milk are run into a beaker with a pipette, and the weight of the beaker and milk are determined. The coil is then intro-



much as possible of the milk is absorbed by the paper. It is often possible to take up almost the last drop of the milk. By then weighing the beaker, the amount of milk absorbed by the coil is determined by difference, and the paper coil is hung up and dried, first in the air and then in the oven, at a temperature not exceeding 100°. Another method of charging the paper coil consists in suspending it by the wire and gradually delivering upon it 5 cc. of the milk from a pipette, the density of the milk being known.



FIG. 34. — The Adams Milk-fat Coil.

The coil containing the dried residue is then transferred to the Soxhlet extraction apparatus (see p. 57) and subjected to continuous extraction with anhydrous ether for at least two hours, the receiving-flask being first accurately weighed. The tared flask with its contents is freed from all remaining ether, first on the water-bath and finally in the air-oven. It is then cooled and weighed, the increase in weight representing the fat in the amount of milk absorbed by the coil. If there is any doubt about all the fat having been extracted at first, the process of extraction may be continued till there is no longer a gain in weight of the flask. Experience soon shows the length of time necessary for the complete extraction, which of course depends on the degree of heat employed, and the frequency with which the extracting-tube overflows. Two hours is ample for most cases, in which the conditions are such that the ether siphons over from the extraction-tube ten times per hour.

FAT METHODS BASED ON CENTRIFUGAL SEPARATION. — These methods are the most practicable for commercial work and for use by the public analyst, since they are much more rapid, and, if carefully carried out, practically as accurate as the Adams method. They all depend upon the use of a centrifugal machine, having hinged pockets in which are carried graduated bottles, into each of which a measured quantity of milk is introduced. The milk is then subjected to the action of a suitable reagent, which dissolves the casein and liberates the fat in a pure state, after which, by whirling at a high speed, the pockets are thrown out horizontally and the milk fat driven into the neck of each bottle, where the amount is directly read.

Various processes of this kind, each having its own special adherents, are in extensive use, among which the best known are the Babcock, the Leffman and Beam, the Gerber, and the Stokes.

A résumé of these processes, showing the reagents employed and other comparative data, is thus tabulated by Allen.*

	Babcock.	Leffman-Beam.	Gerber.	Stokes.
Milk	17.5 cc.	15 cc.	11 cc.	15 cc.
Sulphuric acid, volume	17.5 cc.	9 cc.	10 cc.	13½ cc.
" " specific gravity	1.831 to 1.834	1.85	1.82 to 1.825	1.82 to 1.83
Hydrochloric acid	None	1.5 cc.	None	None
Amyl alcohol	None	1.5 cc.	1.0 cc.	1.5 cc.

The Babcock Process, devised originally for the use of creameries and dairymen, is now extensively employed for fat determination in the laboratory.

It has stood the test of over ten years' successful use in the writer's hands. During this time on various occasions results as determined have been compared with those obtained by the Adams process, and the agreement has been as close as could be expected. The following figures show the results of such comparative determinations made in duplicate on three samples of milk, viz., a whole pure milk, (1) and (2); a watered milk, (3) and (4); and a milk centrifugally skimmed, (5) and (6).

COMPARATIVE FAT DETERMINATION BY ADAMS-SOXHLET AND BY BABCOCK PROCESSES.

		Per Cent of Fat by the Adams-Soxhlet Process.	Per Cent of Fat by the Babcock Process.
A whole milk	(1)	4.27	4.30
	(2)	4.28	4.35
A watered milk	(3)	2.70	2.70
	(4)	2.74	2.80
A skimmed milk	(5)	0.16	0.15
	(6)	0.14	0.15

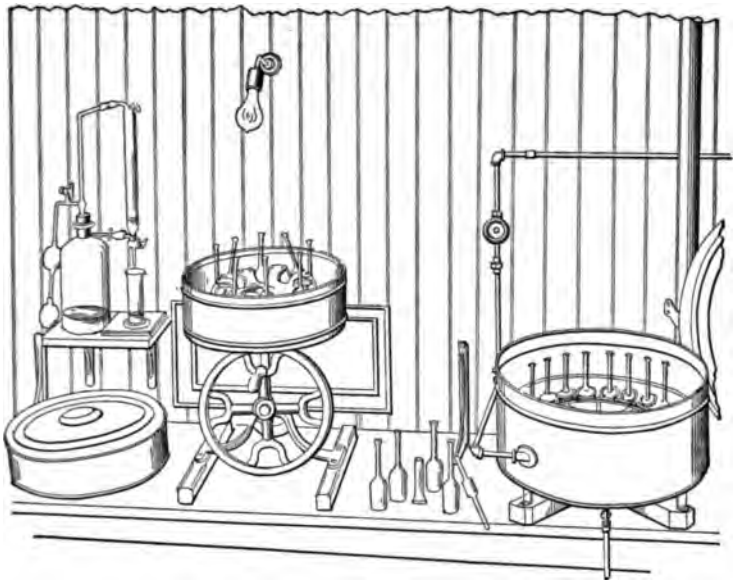


FIG. 35.—Types of the Babcock Centrifuge and Appurtenances. Hand machine at the left; steam-driven machine at the right.

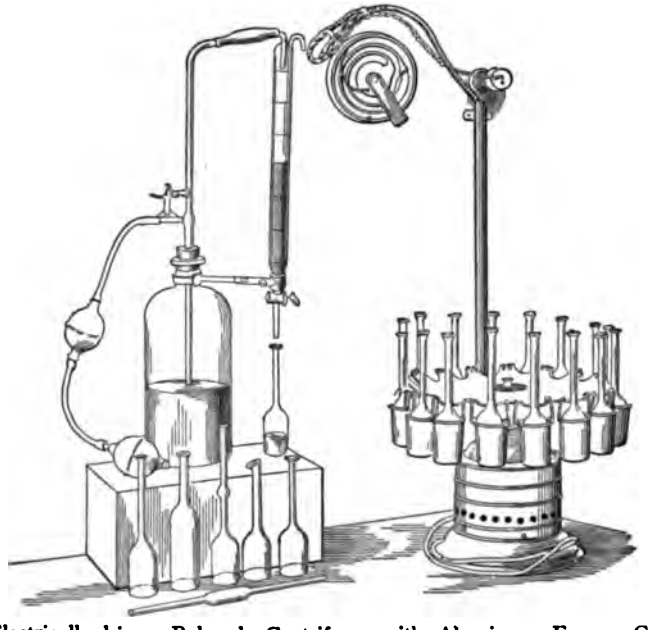


FIG. 36.—Electrically-driven Babcock Centrifuge, with Aluminum Frame, Carrying 16 Bottles. Acid burette at the left.

A sheet-metal safety-shield (removed for showing the construction) normally surrounds the instrument. Such a shield is shown in Fig. 11.

The ordinary Babcock test bottle is shown in Fig. 37, that used for skimmed milk in Fig. 38. The bottles are graduated with reference to using 18 grams of the sample.

Manipulation.—By means of a pipette graduated to hold 17.6 cc. (the average volume of 18 grams) that amount of the thoroughly mixed sample of milk to be tested is transferred to a test bottle, and 17.5 cc. of commercial sulphuric acid of a specific gravity of 1.82 to 1.84 are added



FIG. 37.—Babcock Milk-test Bottle.



FIG. 38.—Babcock Test Bottle for Skimmed Milk.

The Werner-Schmidt Method.—Ten cc. of milk are introduced by means of a pipette into a large test-tube of 50 cc. capacity, and 10 cc. of concentrated hydrochloric acid are added. The mixture is shaken and heated till the liquid turns a dark brown, either by direct boiling for a minute or two, or by immersing the tube in boiling water for from five to ten minutes. The tube is then cooled by immersion in cold water, and 30 cc. of washed ether is added. The tube is closed by a cork provided with tubes similar to a wash-bottle, the larger tube being adapted to slide up and down in the cork, and preferably being turned up slightly at the bottom. The contents of the tube are shaken, the ether layer allowed to separate, and the sliding-tube arranged so that it terminates slightly above the junction of the two layers. The ether is then blown out into a weighed flask. A second and a third portion of ether of 10 cc. each are successively shaken with the acid liquid and added to the contents of the weighed flask, from which the ether is subsequently evaporated and the weight of the fat easily obtained.

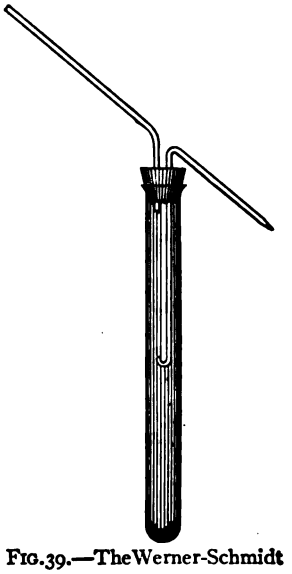


FIG. 39.—The Werner-Schmidt Fat Apparatus.

Instead of measuring the milk into the testing-tube, a known weight of milk may be operated on. A sour milk may be readily tested in this way, provided it is previously well mixed.

Determination of Fat by the Wollny Milk-fat Refractometer.*—This instrument presents the same appearance as the butyro-refractometer, Fig. 91, with an arbitrary scale reading from 0 to 100, the equivalent readings in indices of refraction of the Wollny instrument varying from 1.3332 to 1.4220. Exactly 30 cc. of the milk to be tested are measured into the stoppered flask *A*, Fig. 40. This may be done by the use of the automatic pipette, which holds exactly 7½ cc., removing four pipettes full of the milk. *B* is a numbered tin sampling-tube in which the milk sample is kept for convenience, and into which the automatic pipette readily fits. Having measured 30 cc. of the milk into the flask *A*, 12 drops of a solution of 70 grams potassium bichromate and 312.5 cc. of stronger ammonia in one liter of water may be added as a preservative,

* Milch Zeit., 1900, pp. 50–53.

if the sample is to be kept for some time before finishing the test. Twelve drops of glacial acetic acid are added to curdle the milk. The flask is then corked and shaken for one to two minutes in a mechanical shaker, after which 3 cc. of a standard alkaline solution are added, and the flask corked and shaken for ten minutes in the mechanical shaker, the temperature being kept at 17.5° C. The standard alkaline solution is prepared

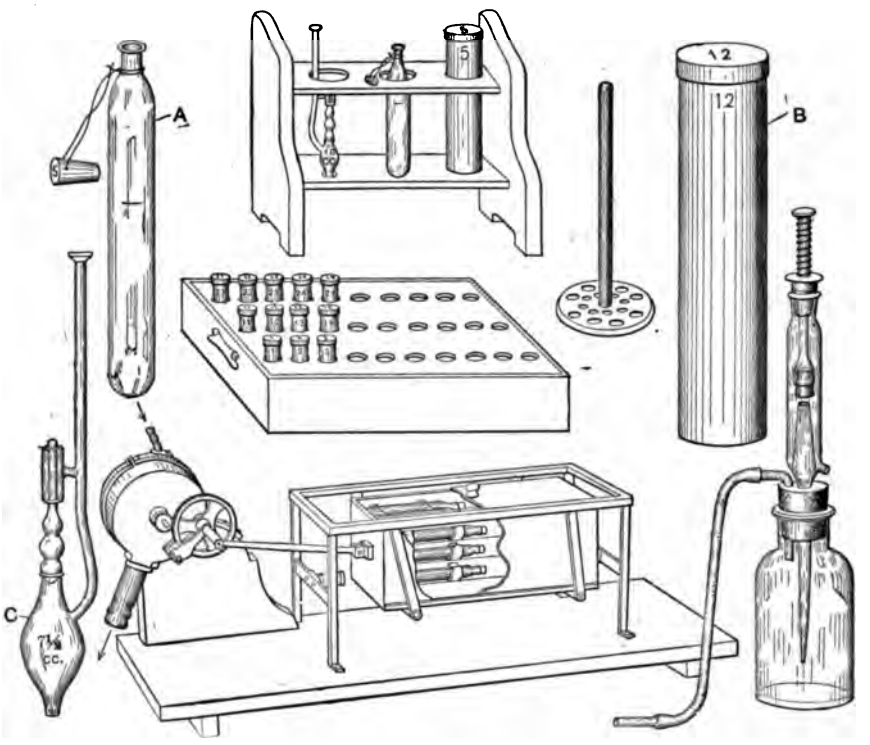


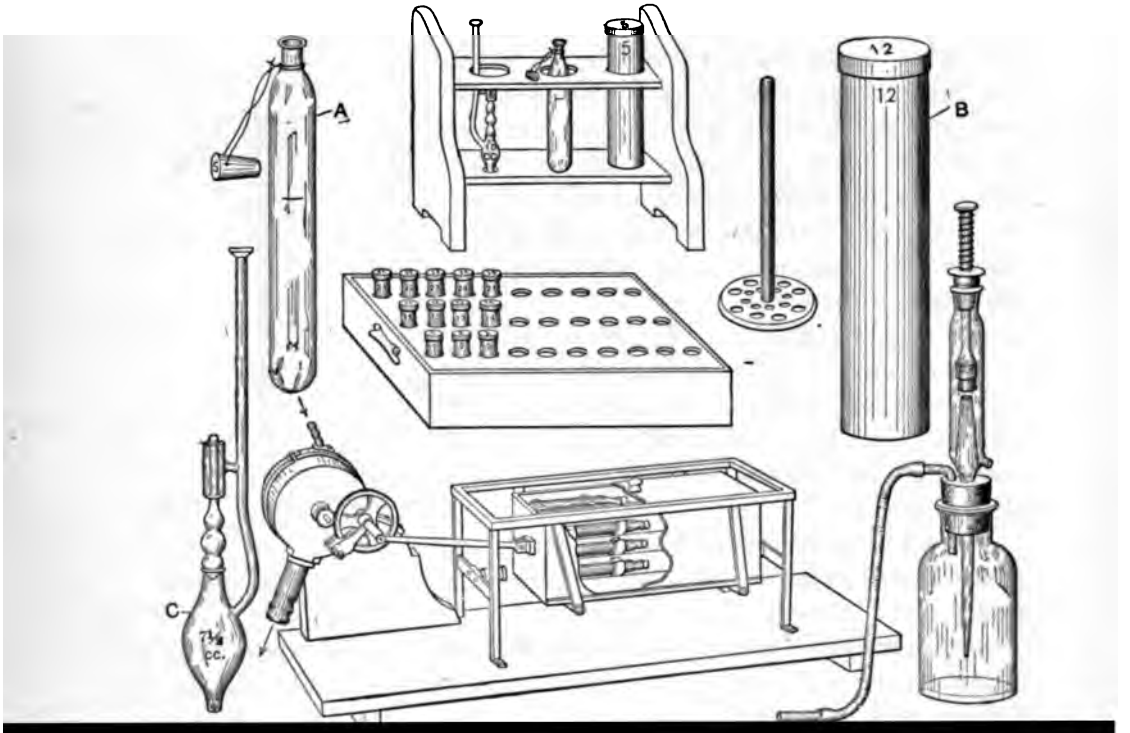
FIG. 40.—Accessories for Carrying Out the Wollney Milk-fat Process.

by dissolving 800 cc. of potassium hydroxide in a liter of water, adding 600 cc. of glycerin and 200 grams pulverized copper hydrate, the mixture being allowed to stand for several days before using, shaking at intervals. Finally 6 cc. of water-saturated ether are added to the mixture in the flask, using for convenience the automatic pipette fitted in the corked bottle as shown. The flask is again shaken for fifteen minutes in the mechanical shaker, and whirled for three minutes in the centrifuge, after which a few drops of the ether solution are transferred to the refractometer, and the reading taken. The percentage of fat is obtained by means of the following table:

PERCENTAGES OF FAT CORRESPONDING TO SCALE READINGS ON THE WOLLNY REFRACTOMETER.

Scale Reading.	Per Cent Fat.	Scale Reading.	Per Cent Fat.	Scale Reading.	Per Cent Fat.	Scale Reading.	Per Cent Fat.	Scale Reading.	Per Cent Fat.	Scale Reading.	Per Cent Fat.
20.0	.....	24.5	0.41	29.0	0.87	33.5	1.34	38.0	1.85	42.5	2.41
1	.....	6	0.42	1	0.88	6	1.35	1	1.87	6	2.43
2	.....	7	0.43	2	0.89	7	1.36	2	1.88	7	2.44
3	.....	8	0.44	3	0.90	8	1.37	3	1.89	8	2.46
4	.....	9	0.45	4	0.91	9	1.38	4	1.90	9	2.47
5	.....	25.0	0.46	5	0.92	34.0	1.39	5	1.91	43.0	2.49
6	0.00	1	0.47	6	0.93	1	1.40	6	1.92	1	2.50
7	0.01	2	0.48	7	0.94	2	1.42	7	1.93	2	2.51
8	0.02	3	0.49	8	0.95	3	1.43	8	1.94	3	2.52
9	0.03	4	0.50	9	0.96	4	1.44	9	1.95	4	2.54
21.0	0.04	5	0.51	30.0	0.97	5	1.45	39.0	1.96	5	2.55
1	0.05	6	0.52	1	0.98	6	1.46	1	1.98	6	2.56
2	0.06	7	0.53	2	0.99	7	1.47	2	1.99	7	2.58
3	0.08	8	0.54	3	1.00	8	1.48	3	2.00	8	2.60
4	0.09	9	0.55	4	1.01	9	1.49	4	2.02	9	2.61
5	0.10	26.0	0.57	5	1.02	35.0	1.50	5	2.03	44.0	2.63
6	0.11	1	0.58	6	1.03	1	1.51	6	2.04	1	2.64
7	0.12	2	0.59	7	1.04	2	1.52	7	2.05	2	2.65
8	0.13	3	0.60	8	1.05	3	1.54	8	2.07	3	2.67
9	0.14	4	0.61	9	1.06	4	1.55	9	2.08	4	2.68
22.0	0.15	5	0.62	31.0	1.07	5	1.56	40.0	2.09	5	2.70
1	0.16	6	0.63	1	1.08	6	1.57	1	2.10	6	2.71
2	0.17	7	0.64	2	1.09	7	1.58	2	2.12	7	2.72
3	0.18	8	0.65	3	1.10	8	1.59	3	2.13	8	2.74
4	0.19	9	0.66	4	1.11	9	1.60	4	2.14	9	2.75
5	0.20	27.0	0.67	5	1.12	36.0	1.61	5	2.15	45.0	2.77
6	0.21	1	0.68	6	1.13	1	1.62	6	2.16	1	2.78
7	0.22	2	0.69	7	1.14	2	1.64	7	2.18	2	2.79
8	0.23	3	0.70	8	1.15	3	1.65	8	2.20	3	2.80
9	0.24	4	0.71	9	1.16	4	1.66	9	2.21	4	2.82
23.0	0.25	5	0.72	32.0	1.17	5	1.67	41.0	2.23	5	2.84
1	0.26	6	0.73	1	1.18	6	1.68	1	2.24	6	2.85
2	0.27	7	0.74	2	1.19	7	1.69	2	2.25	7	2.87
3	0.28	8	0.75	3	1.20	8	1.70	3	2.26	8	2.88
4	0.29	9	0.76	4	1.22	9	1.71	4	2.27	9	2.89
5	0.30	28.0	0.77	5	1.23	37.0	1.72	5	2.28	46.0	2.90
6	0.31	1	0.78	6	1.24	1	1.73	6	2.30	1	2.92
7	0.32	2	0.79	7	1.25	2	1.75	7	2.32	2	2.93
8	0.33	3	0.80	8	1.26	3	1.76	8	2.33	3	2.94
9	0.34	4	0.81	9	1.27	4	1.78	9	2.34	4	2.96
24.0	0.36	5	0.82	33.0	1.28	5	1.79	42.0	2.35	5	2.98
1	0.37	6	0.83	1	1.29	6	1.80	1	2.37	6	3.00
2	0.38	7	0.84	2	1.30	7	1.81	2	2.38	7	3.01
3	0.39	8	0.85	3	1.31	8	1.82	3	2.39	8	3.02
4	0.40	9	0.86	4	1.32	9	1.84	4	2.40	9	3.03
5	0.41	29.0	0.87	5	1.34	38.0	1.85	5	2.41	47.0	3.05

if the sample is to be kept for some time before finishing the test. Twelve drops of glacial acetic acid are added to curdle the milk. The flask is then corked and shaken for one to two minutes in a mechanical shaker, after which 3 cc. of a standard alkaline solution are added, and the flask corked and shaken for ten minutes in the mechanical shaker, the temperature being kept at 17.5° C. The standard alkaline solution is prepared



PERCENTAGES OF FAT CORRESPONDING TO SCALE READINGS ON THE WOLLNY REFRACTOMETER.

Scale Reading.	Per Cent Fat.	Scale Reading.	Per Cent Fat.	Scale Reading.	Per Cent Fat.	Scale Reading.	Per Cent Fat.	Scale Reading.	Per Cent Fat.	Scale Reading.	Per Cent Fat.
20.0	.....	24.5	0.41	29.0	0.87	33.5	1.34	38.0	1.85	42.5	2.41
1	.....	6	0.42	1	0.88	6	1.35	1	1.87	6	2.43
2	.....	7	0.43	2	0.89	7	1.36	2	1.88	7	2.44
3	.....	8	0.44	3	0.90	8	1.37	3	1.89	8	2.46
4	.....	9	0.45	4	0.91	9	1.38	4	1.90	9	2.47
5	.....	25.0	0.46	5	0.92	34.0	1.39	5	1.91	43.0	2.49
6	0.00	1	0.47	6	0.93	1	1.40	6	1.92	1	2.50
7	0.01	2	0.48	7	0.94	2	1.42	7	1.93	2	2.51
8	0.02	3	0.49	8	0.95	3	1.43	8	1.94	3	2.52
9	0.03	4	0.50	9	0.96	4	1.44	9	1.95	4	2.54
21.0	0.04	5	0.51	30.0	0.97	5	1.45	39.0	1.96	5	2.55
1	0.05	6	0.52	1	0.98	6	1.46	1	1.98	6	2.56
2	0.06	7	0.53	2	0.99	7	1.47	2	1.99	7	2.58
3	0.08	8	0.54	3	1.00	8	1.48	3	2.00	8	2.60
4	0.09	9	0.55	4	1.01	9	1.49	4	2.02	9	2.61
5	0.10	26.0	0.57	5	1.02	35.0	1.50	5	2.03	44.0	2.63
6	0.11	1	0.58	6	1.03	1	1.51	6	2.04	1	2.64
7	0.12	2	0.59	7	1.04	2	1.52	7	2.05	2	2.65
8	0.13	3	0.60	8	1.05	3	1.54	8	2.07	3	2.67
9	0.14	4	0.61	9	1.06	4	1.55	9	2.08	4	2.68
22.0	0.15	5	0.62	31.0	1.07	5	1.56	40.0	2.09	5	2.70
1	0.16	6	0.63	1	1.08	6	1.57	1	2.10	6	2.71
2	0.17	7	0.64	2	1.09	7	1.58	2	2.12	7	2.72
3	0.18	8	0.65	3	1.10	8	1.59	3	2.13	8	2.74
4	0.19	9	0.66	4	1.11	9	1.60	4	2.14	9	2.75
5	0.20	27.0	0.67	5	1.12	36.0	1.61	5	2.15	45.0	2.77
6	0.21	1	0.68	6	1.13	1	1.62	6	2.16	1	2.78
7	0.22	2	0.69	7	1.14	2	1.64	7	2.18	2	2.79
8	0.23	3	0.70	8	1.15	3	1.65	8	2.20	3	2.80
9	0.24	4	0.71	9	1.16	4	1.66	9	2.21	4	2.82
23.0	0.25	5	0.72	32.0	1.17	5	1.67	41.0	2.23	5	2.84
1	0.26	6	0.73	1	1.18	6	1.68	1	2.24	6	2.85
2	0.27	7	0.74	2	1.19	7	1.69	2	2.25	7	2.87
3	0.28	8	0.75	3	1.20	8	1.70	3	2.26	8	2.88
4	0.29	9	0.76	4	1.22	9	1.71	4	2.27	9	2.89
5	0.30	28.0	0.77	5	1.23	37.0	1.72	5	2.28	46.0	2.90
6	0.31	1	0.78	6	1.24	1	1.73	6	2.30	1	2.92
7	0.32	2	0.79	7	1.25	2	1.75	7	2.32	2	2.93
8	0.33	3	0.80	8	1.26	3	1.76	8	2.33	3	2.94
9	0.34	4	0.81	9	1.27	4	1.78	9	2.34	4	2.96
24.0	0.36	5	0.82	33.0	1.28	5	1.79	42.0	2.35	5	2.98
1	0.37	6	0.83	1	1.29	6	1.80	1	2.37	6	3.00
2	0.38	7	0.84	2	1.30	7	1.81	2	2.38	7	3.01
3	0.39	8	0.85	3	1.31	8	1.82	3	2.39	8	3.02
4	0.40	9	0.86	4	1.32	9	1.84	4	2.40	9	3.03
5	0.41	29.0	0.87	5	1.34	38.0	1.85	5	2.41	47.0	3.05

PERCENTAGES OF FAT CORRESPONDING TO SCALE READINGS ON THE WOLLNY REFRACTOMETER—(*Continued*).

Scale Reading.	Per Cent Fat.	Scale Reading.	Per Cent Fat.	Scale Reading.	Per Cent Fat.	Scale Reading.	Per Cent Fat.	Scale Reading.	Per Cent Fat.	Scale Reading.	Per Cent Fat.
47.0	3.05	50.5	3.59	54.0	4.18	57.5	4.78	61.0	5.44	64.5	6.14
1	3.06	6	3.60	1	4.20	6	4.80	1	5.46	6	6.16
2	3.08	7	3.61	2	4.22	7	4.82	2	5.48	7	6.18
3	3.10	8	3.63	3	4.23	8	4.84	3	5.50	8	6.20
4	3.12	9	3.64	4	4.25	9	4.86	4	5.52	9	6.22
5	3.14	51.0	3.66	5	4.26	58.0	4.88	5	5.54	65.0	6.24
6	3.15	1	3.67	6	4.28	1	4.90	6	5.56	1	6.27
7	3.16	2	3.68	7	4.29	2	4.92	7	5.58	2	6.29
8	3.17	3	3.70	8	4.31	3	4.94	8	5.60	3	6.31
9	3.18	4	3.72	9	4.33	4	4.95	9	5.61	4	6.34
48.0	3.20	5	3.74	55.0	4.35	5	4.97	62.0	5.63	5	6.36
1	3.21	6	3.76	1	4.37	6	4.98	1	5.65	6	6.38
2	3.23	7	3.78	2	4.38	7	5.00	2	5.66	7	6.40
3	3.25	8	3.80	3	4.40	8	5.02	3	5.68	8	6.42
4	3.27	9	3.82	4	4.42	9	5.04	4	5.70	9	6.44
5	3.28	52.0	3.84	5	4.43	59.0	5.06	5	5.72	66.0	6.46
6	3.30	1	3.85	6	4.44	1	5.08	6	5.74		
7	3.32	2	3.87	7	4.46	2	5.10	7	5.76		
8	3.33	3	3.89	8	4.48	3	5.11	8	5.78		
9	3.34	4	3.90	9	4.49	4	5.13	9	5.80		
49.0	3.36	5	3.92	56.0	4.51	5	5.15	63.0	5.82		
1	3.38	6	3.93	1	4.53	6	5.17	1	5.84		
2	3.40	7	3.95	2	4.55	7	5.19	2	5.86		
3	3.42	8	3.97	3	4.57	8	5.20	3	5.88		
4	3.43	9	3.99	4	4.59	9	5.22	4	5.90		
5	3.44	53.0	4.01	5	4.60	60.0	5.24	5	5.92		
6	3.45	1	4.03	6	4.61	1	5.26	6	5.94		
7	3.46	2	4.04	7	4.63	2	5.28	7	5.96		
8	3.48	3	4.06	8	4.65	3	5.30	8	5.98		
9	3.50	4	4.07	9	4.67	4	5.32	9	6.00		
50.0	3.51	5	4.09	57.0	4.69	5	5.34	64.0	6.02		

INDICES OF REFRACTION (n_D) CORRESPONDING TO SCALE READINGS OF THE WOLLNY MILK-FAT REFRACTOMETER.

Refractive Index. n_D.	Fourth Decimal of n_D.									
	0	1	2	3	4	5	6	7	8	9
	Scale Readings.									
1.333	.....	.....	0.0	0.1	0.2	0.3	0.4	0.5	0.5	0.6
1.334	0.7	0.8	0.9	1.0	1.1	1.2	1.3	1.4	1.5	1.6
1.335	1.7	1.8	1.9	2.0	2.1	2.1	2.2	2.3	2.4	2.5
1.336	2.8	2.7	2.8	2.9	3.0	3.1	3.2	3.3	3.4	3.5
1.337	3.6	3.7	3.7	3.8	3.9	4.0	4.1	4.2	4.3	4.4
1.338	4.5	4.6	4.7	4.8	4.9	5.0	5.1	5.2	5.3	5.4
1.339	5.5	5.6	5.7	5.8	5.9	6.0	6.1	6.2	6.3	6.4
1.340	6.5	6.6	6.7	6.8	6.9	6.9	7.0	7.1	7.2	7.3
1.341	7.4	7.5	7.6	7.7	7.8	7.9	8.0	8.1	8.2	8.3
1.342	8.4	8.5	8.6	8.7	8.8	8.9	9.0	9.1	9.2	9.3
1.343	9.4	9.5	9.6	9.7	9.8	9.9	10.0	10.1	10.2	10.3
1.344	10.4	10.5	10.6	10.7	10.8	10.9	11.0	11.1	11.2	11.3
1.345	11.4	11.5	11.5	11.6	11.7	11.8	11.9	12.0	12.1	12.2
1.346	12.3	12.4	12.5	12.6	12.7	12.8	12.9	13.0	13.1	13.2
1.347	13.3	13.4	13.5	13.6	13.7	13.8	13.9	14.0	14.1	14.2
1.348	14.3	14.4	14.5	14.6	14.7	14.8	14.9	15.0	15.1	15.2
1.349	15.3	15.4	15.5	15.6	15.7	15.8	15.9	16.0	16.1	16.2
1.350	16.3	16.4	16.5	16.6	16.7	16.8	16.9	17.0	17.1	17.2
1.351	17.3	17.4	17.5	17.6	17.7	17.8	17.9	18.0	18.1	18.2
1.352	18.3	18.4	18.5	18.6	18.7	18.8	18.9	19.0	19.1	19.2
1.353	19.3	19.4	19.5	19.6	19.7	19.8	19.9	20.0	20.1	20.2
1.354	20.3	20.4	20.5	20.6	20.7	20.8	20.9	21.0	21.1	21.2
1.355	21.3	21.4	21.5	21.6	21.7	21.8	21.9	22.0	22.1	22.2
1.356	22.3	22.4	22.5	22.6	22.7	22.8	22.9	23.0	23.1	23.2
1.357	23.3	23.4	23.5	23.6	23.7	23.8	23.9	24.0	24.1	24.2
1.358	24.3	24.4	24.5	24.6	24.7	24.8	24.9	25.0	25.1	25.2
1.359	25.3	25.4	25.5	25.6	25.7	25.8	25.9	26.0	26.1	26.2
1.360	26.3	26.4	26.5	26.6	26.7	26.8	26.9	27.0	27.1	27.3
1.361	27.4	27.5	27.6	27.7	27.8	27.9	28.0	28.1	28.2	28.3
1.362	28.4	28.5	28.6	28.7	28.8	28.9	29.0	29.1	29.2	29.3
1.363	29.4	29.5	29.6	29.7	29.8	29.9	30.0	30.1	30.2	30.3
1.364	30.4	30.5	30.6	30.7	30.8	31.0	31.1	31.2	31.3	31.4
1.365	31.5	31.6	31.7	31.8	31.9	32.0	32.1	32.2	32.3	32.4
1.366	32.5	32.7	32.8	32.9	33.0	33.1	33.2	33.3	33.4	33.5
1.367	33.6	33.7	33.8	33.9	34.0	34.2	34.3	34.4	34.5	34.6
1.368	34.7	34.8	34.9	35.0	35.1	35.2	35.3	35.4	35.5	35.6
1.369	35.7	35.8	36.0	36.1	36.2	36.3	36.4	36.5	36.6	36.7
1.370	36.8	36.9	37.0	37.1	37.2	37.3	37.4	37.6	37.7	37.8
1.371	37.9	38.0	38.1	38.2	38.3	38.4	38.5	38.6	38.7	38.8
1.372	38.9	39.0	39.2	39.3	39.4	39.5	39.6	39.7	39.8	39.9
1.373	40.0	40.1	40.2	40.3	40.4	40.5	40.7	40.8	40.9	41.0
1.374	41.1	41.2	41.3	41.4	41.5	41.6	41.8	41.9	42.0	42.1
1.375	42.2	42.3	42.4	42.5	42.6	42.7	42.8	42.9	43.0	43.1
1.376	43.2	43.3	43.4	43.6	43.7	43.8	43.9	44.0	44.1	44.2
1.377	44.3	44.4	44.6	44.7	44.8	44.9	45.0	45.1	45.2	45.3
1.378	45.4	45.6	45.7	45.8	45.9	46.0	46.1	46.2	46.3	46.4
1.379	46.6	46.7	46.8	46.9	47.0	47.1	47.2	47.3	47.4	47.6

INDICES OF REFRACTION (n_D) CORRESPONDING TO SCALE READINGS OF THE WOLLNY MILK-FAT REFRACTOMETER—(*Continued*).

Refractive Index, n_D.	Fourth Decimal of n_D.									
	0	1	2	3	4	5	6	7	8	9
	Scale Readings.									
1.380	47.7	47.8	47.9	48.0	48.1	48.2	48.3	48.4	48.6	48.7
1.381	48.8	48.9	49.0	49.1	49.2	49.3	49.4	49.6	49.7	49.8
1.382	49.9	50.0	50.1	50.2	50.3	50.4	50.6	50.7	50.8	50.9
1.383	51.0	51.1	51.2	51.3	51.4	51.6	51.7	51.8	51.9	52.0
1.384	52.1	52.2	52.3	52.4	52.6	52.7	52.8	52.9	53.0	53.1
1.385	53.2	53.3	53.4	53.6	53.7	53.8	53.9	54.0	54.1	54.2
1.386	54.3	54.4	54.6	54.7	54.8	54.9	55.0	55.1	55.2	55.3
1.387	55.4	55.6	55.7	55.8	55.9	56.0	56.1	56.2	56.3	56.5
1.388	56.6	56.7	56.8	56.9	57.1	57.2	57.3	57.4	57.6	57.7
1.389	57.8	57.9	58.0	58.1	58.2	58.3	58.4	58.6	58.7	58.8
1.390	58.9	59.0	59.1	59.2	59.4	59.5	59.6	59.8	59.9	60.0
1.391	60.1	60.2	60.3	60.4	60.6	60.7	60.8	60.9	61.0	61.1
1.392	61.3	61.4	61.5	61.6	61.8	61.9	62.0	62.1	62.2	62.3
1.393	62.4	62.6	62.7	62.8	62.9	63.0	63.2	63.3	63.4	63.5
1.394	63.6	63.8	63.9	64.0	64.1	64.2	64.4	64.5	64.6	64.7
1.395	64.8	65.0	65.1	65.2	65.3	65.4	65.6	65.7	65.8	65.9
1.396	66.0	66.2	66.3	66.4	66.5	66.6	66.8	66.9	67.0	67.1
1.397	67.2	67.4	67.5	67.6	67.7	67.8	67.9	68.1	68.2	68.3
1.398	68.4	68.6	68.7	68.8	68.9	69.0	69.1	69.3	69.4	69.5
1.399	69.6	69.8	69.9	70.0	70.1	70.2	70.4	70.5	70.6	70.8
1.400	70.9	71.0	71.1	71.2	71.4	71.5	71.6	71.8	71.9	72.0
1.401	72.1	72.2	72.4	72.5	72.6	72.8	72.9	73.0	73.1	73.2
1.402	73.4	73.5	73.6	73.8	73.9	74.0	74.1	74.2	74.4	74.5
1.403	74.6	74.8	74.9	75.0	75.1	75.2	75.4	75.5	75.6	75.8
1.404	75.9	76.0	76.1	76.2	76.4	76.5	76.6	76.8	76.9	77.0
1.405	77.1	77.2	77.4	77.5	77.7	77.8	77.9	78.1	78.2	78.3
1.406	78.5	78.6	78.7	78.8	79.0	79.1	79.2	79.4	79.5	79.6
1.407	79.8	79.9	80.0	80.1	80.2	80.4	80.5	80.6	80.8	80.9
1.408	81.0	81.1	81.2	81.4	81.5	81.6	81.7	81.9	82.0	82.1
1.409	82.3	82.4	82.5	82.6	82.8	82.9	83.0	83.2	83.3	83.4

DETERMINATION OF PROTEIDS.—For determination of the total nitrogen in milk, 5 cc. are measured direct into a Kjeldahl digestion-flask, or a known weight from a weighing-bottle may be used, and the regular Gunning method is employed as described on page 61, proceeding with the digestion at once without evaporation.

The total nitrogen, multiplied by 6.38, gives the total proteids. By many the old factor of 6.25 is still employed, but in view of the fact that both casein and albumin have been found to contain 15.7% of nitrogen, there would seem to be the best reasons for employing 6.38 as a factor $\left(\frac{100}{15.7}\right)$.

Ritthausen's Method.—Ten grams of milk are measured into a beaker and diluted with water to about 100 cc. Five cc. of a solution of copper sulphate (strength of Fehling's copper solution, 34.64 grams $CuSO_4$ in 500 cc. of water) are added and the mixture stirred. A solution of sodium hydroxide (25 grams to the liter) is added cautiously a little at a time, till the liquid is nearly, but not quite neutral, avoiding an excess of alkali, as this would prevent the complete precipitation of the proteids. Allow the precipitate to settle, and pour off the supernatant liquid through a weighed filter, previously dried at 130° C. Wash a number of times by decantation, and transfer the precipitate to the filter, being careful to remove the portions adhering to the sides of the beaker with a rubber-tipped rod. Wash thoroughly with water, and drain dry, after which the precipitate is washed with strong alcohol, dried, extracted with ether, preferably in a Soxhlet extractor, and then transferred on the filter to the oven, dried at 130° C., and weighed. The filter and precipitate are then burnt to an ash in a porcelain crucible, and the weight of the residue subtracted from the first weight gives that of the proteids.

Richmond * recommends modifying this process to the extent of neutralizing the milk, using phenolphthalein as an indicator, before adding the copper sulphate solution, and using only 2.5 cc. of the latter.

Determination of Casein.—*Official Method of the A. O. A. C.*—Ten grams of the milk are placed in a beaker, and made up with water to about 100 cc. at 40° to 42° C. One and one-half cc. of a 10% solution (by weight) of acetic acid are added, the mixture stirred, warmed to the above temperature, and allowed to stand for from three to five minutes, till a flocculent precipitate separates, leaving a clear supernatant liquid. Decant

* Dairy Chem., p. 107.

upon a filter, wash with cold water two or three times by decantation, and finally transfer the whole of the precipitate to the filter, and, after filtering, wash two or three times. The filtrate should be clear or nearly so. If not, it can generally be made so by repeated filtrations, and the washing done afterwards. The filter containing the washed precipitate is transferred to the Kjeldahl digestion-flask and the nitrogen obtained by the Gunning process. $N \times 6.38 = \text{casein}$.

Provisional Magnesium Sulphate Method.—A solution of magnesium sulphate, saturated at from 40° to 45°, is prepared, and 50 cc. are added to 5 grams of milk, the mixture being heated to about 45° till the precipitate forms and settles out, leaving a clear supernatant liquid. The liquid is first decanted through a filter, and the precipitate collected thereon and washed several times with the above saturated magnesium sulphate solution, the temperature being kept at about 45° during the operation. The precipitate, with filter, is transferred to a digestion-flask, and the nitrogen determined.

Determination of Albumin.—*Optional Methods of the A. O. A. C.*—To the filtrate from the direct determination of casein by the magnesium sulphate method as above described, or to the filtrate from the acetic acid method neutralized with sodium hydroxide, 0.3 cc. of a 10% solution of acetic acid is added, and the mixture is boiled till the albumin is completely precipitated. The precipitate is collected on a filter and washed, the nitrogen being determined in the precipitate, and the factor 6.38 * used in calculating the albumin therefrom.

Leffman and Beam's Modified Method for Albumin and Casein.—Owing to the tedious processes of washing and filtering incidental to the above methods for determining casein, the following is suggested. Twenty cc. of the milk are mixed with saturated magnesium sulphate solution, and



Determination of Nitrogen as Caseoses, Amido-compounds, Peptones, and Ammonia.—Van Slyke* proceeds as follows: The filtrate from the determination of the albumin, page 110, is heated to 70° C., 1 cc. of 50% sulphuric acid is first added, and afterwards chemically pure zinc sulphate to saturation. The mixture is allowed to stand at 70° until the caseoses separate out and settle. Cool, filter, wash with a saturated zinc sulphate solution slightly acidified with sulphuric acid, and determine the nitrogen of the caseoses in the precipitate.

For Amido-compounds and Ammonia treat 50 grams of the milk in a 250-cc. graduated flask with 1 gram sodium chloride and a 12% solution of tannin, added drop by drop till no further precipitate is formed. Dilute to the 250-cc. mark, shake, and filter. Determine the nitrogen in 50 cc. of the filtrate, the result being the combined nitrogen of the amido-compounds and ammonia.

Distil with magnesium oxide 100 cc. of the filtrate from the tannin salt solution, receiving the distillate in a standardized acid, and titrating in the usual way for the ammonia.

Calculate the nitrogen of the *peptones* by subtracting from the total nitrogen that due to all other forms.

Van Slyke has furnished the following unpublished analysis of a sample of milk three months old, kept under antiseptic conditions by chloroform.

Per Cent Total N.	Per Cent Sol. Nitrogen.	Per Cent N as Paranuclein, Caseoses, and Peptones.	Per Cent N as Amides.
0.561	0.099	0.074	0.025

DETERMINATION OF MILK SUGAR.—If a polariscope is available, the sugar of milk can most readily and conveniently be determined by optical methods. In the absence of a polariscope, the reducing power of milk sugar on copper salts may be utilized quite accurately in determining the sugar, using either volumetric or gravimetric methods as desired.

Determination by Optical Methods.—REAGENTS.—*Acid Nitrate of Mercury.*—This solution is prepared by dissolving metallic mercury in twice its weight of nitric acid of specific gravity 1.42, and adding to the solution an equal volume of water. One cc. of this reagent will be

* N. Y. Exp. Station, Bul. 215, p. 102.

found sufficient to precipitate the proteids and fat completely from 65 grams of milk, but if more is employed the result of the analysis is not affected.

Mercuric Iodide Solution.—33.2 grams of potassium iodide are mixed with 13.5 grams of mercuric chloride, 20 cc. of acetic acid, and 640 cc. of water.

Subacetate of Lead Solution, U. S. P. See p. 481.

NOTES.—For the Laurent polariscope, in which the normal weight for sucrose is 16.19 grams, the corresponding normal weight for lactose is 20.496, while for the Soleil-Ventzke instrument, in which the sucrose normal weight is 26.048 grams, the corresponding lactose normal weight is 32.975.*

It is customary to employ three times the normal weight of milk in the case of the Laurent instrument (viz., 61.48 grams) and twice the normal weight in the case of the Soleil-Ventzke (viz., 65.95 grams).

As it is more convenient to measure the milk than to weigh it, and as the volume varies with the specific gravity, the following table is useful, showing the quantity to be measured in any case, having first determined the specific gravity.

Specific Gravity.	Volume of Milk to be Used.	
	For Polariscopes of which the Sucrose Normal Weight is 16.19 Grams.	For Polariscopes of which the Sucrose Normal Weight is 26.048 Grams.
1.024	60.0 cc.	64.4 cc.
1.026	59.9 cc.	64.3 cc.
1.028	50.8 cc.	64.15 cc.
1.030	59.7 cc.	64.0 cc.
1.032	59.6 cc.	63.9 cc.
1.034	59.5 cc.	63.8 cc.
1.035	59.35 cc.	63.7 cc.

water. Then add from a pipette enough water to make up for the volume of the precipitated proteids and fat, insuring 100 cc. of sugar solution. If the Laurent instrument is used, the amount added as prescribed by the A. O. A. C. is 2.4 cc., and with the Soleil-Ventzke 2.6 cc. The contents of the flask are then shaken and poured upon a dry filter. The filtrate, which should be perfectly clear, is polarized in a 200-mm. tube, and the reading, divided by 3 for the Laurent and by 2 for the Soleil-Ventzke, gives the percentage of lactose directly.

Allowance for the Volume of the Precipitate.—This of course varies with the content in proteids and fat, and while the above allowance gives in most cases sufficiently close results, it is not exact. Leffman and Beam * advise that the amount of water to be added above 100 cc. be calculated in each case from the percentage of proteids and fat previously found by analysis, multiplying the actual weight of the fat in grams in the sample taken by 1.075, and the weight of proteids by 0.8, the sum of the two results being the volume in cubic centimeters occupied by the precipitate.

All the calculations are avoided by employing the double-dilution method, which is to be recommended when very particular results are required.

Wiley and Ewell's Double-dilution Method.†—Two flasks are employed graduated at 100 and 200 cc. respectively, into each of which are introduced 65.95 grams of milk, if the Soleil-Ventzke instrument is used (or 61.48 grams in case the Laurent is used) and 4 cc. of the mercuric nitrate solution are added, both flasks being filled to the mark and shaken. The contents are filtered and the polarization is made in each case in a 400-mm. tube.

The second reading (that of the more dilute solution) is multiplied by 2, and the product subtracted from the first reading; the remainder is then multiplied by 2, and the product subtracted from the first reading (that of the stronger or 100 cc. solution). The result is the corrected reading, which, divided by 4, gives the exact per cent of milk sugar in the sample. This method depends on the fact that within ordinary limits the polarizations of two solutions of the same substance are inversely proportional to their volumes.

DETERMINATION OF MILK SUGAR BY FEHLING'S SOLUTION.—Twenty-five grams of the milk (24.2 cc.) are transferred to a 250-cc. flask, 0.5 cc. of

* Milk and Milk Products, p. 38.

† Wiley's Agricultural Analysis, p. 278; Analyst, 21, 1896, p. 182.

a 30% solution of acetic acid are added and the contents well shaken. After standing for a few minutes, about 100 cc. of boiling water are run in, the contents again shaken, 25 cc. of alumina cream are next added, the flask shaken once more, and set aside for at least ten minutes. The supernatant liquid is then poured upon a previously wetted ribbed filter, and finally the whole contents of the flask are brought thereon, and the filtrate and washings made up to 250 cc. The filtrate must be perfectly clear. The milk sugar in a solution thus precipitated would ordinarily not exceed ½ of 1 per cent.

Volumetric Fehling Process.—From a burette containing the clear milk sugar solution above prepared, run a measured volume into the boiling Fehling liquor containing 5 cc. each of copper and alkali solution till sufficient has been introduced to completely reduce the copper, conducting the operation in the manner described in detail on page 486.

As 0.067 gram of milk sugar will reduce 10 cc. of Fehling solution (see p. 488), it follows that the number of cubic centimeters of sugar-containing solution required for making the test (using preferably the average of several determinations) will contain 0.067 gram of milk sugar, from which the percentage is readily computed. Thus if 16 cc. of the milk sugar solution are necessary to reduce the copper, then 16 cc. contain 0.067 gram milk sugar.

250 cc. of solution contain 25 grams milk,
1 cc. " " " 0.1 " "
16 cc. " " " 1.6 " "

and 1.6 grams milk contain 0.067 gram milk sugar. Therefore the sample contains $\frac{.067 \times 100}{1.6} = 4.19\%$.

Gravimetric Fehling Processes.—O'Sullivan Defren Method.—Twenty-

tion are added. Then 8.8 cc. of half-normal sodium hydroxide are run in, or a sufficient quantity to nearly but not quite neutralize, the solution being still slightly acid. The flask is filled to the mark, shaken, and the contents filtered, using a dry filter.

One hundred cc. of the filtrate are added to 50 cc. of the mixed Fehling solution, which is boiled briskly in a beaker (using 25 cc. each of the copper and alkali solution). After boiling for six minutes, filter rapidly through a Gooch crucible provided with a layer of asbestos as described on page 489, and wash with boiling water till free from alkali. The asbestos film with the adhering cuprous oxide is washed into a beaker by hot dilute nitric acid, and after complete solution of the copper is assured, it is again filtered and washed with hot water till a clean solution containing all the copper is obtained. Add 10 cc. of dilute sulphuric acid (containing 200 cc. of sulphuric acid, specific gravity 1.84 per liter) and evaporate on the steam-bath till the copper has largely crystallized, then carefully continue the heating over a hot plate till the nitric acid is driven out, as evidenced by the white fumes of sulphuric. Add 8 or 10 drops nitric acid (specific gravity 1.42) and rinse into a *very clean* tared platinum dish of about 100 cc. capacity, in which the copper is deposited by electrolysis. See page 493.

The weight of milk sugar is determined from that of copper found, from the table on page 116.

Relation between Specific Gravity, Fat, and Total Solids of Milk.— The close relationship existing between these factors has long been known, and many formulæ have been devised, whereby, if two of them are known, the third may be computed with considerable approach to accuracy. The specific gravity and the fat are very readily determined by any dairyman, by the aid of a lactometer and the Babcock apparatus. The total solids are ascertained with more difficulty, since the use of more involved and costly apparatus is necessary, besides considerable technical skill. It is therefore common for producers to calculate the total solids from the fat and specific gravity, using one of the many tables prepared for the purpose, based on some one of the best accepted formulæ. The total solids can thus be calculated to within two or three tenths of a per cent.

The two most commonly used formulæ for this purpose are those of Hehner and Richmond in England, and Babcock in the United States. Hehner and Richmond's formula is

$$T = 0.25S + 1.2F + 0.14,$$

SOXHLET'S TABLE FOR THE DETERMINATION OF LACTOSE.*

Milligrams of Copper.	Milligrams of Lactose.	Milligrams of Copper.	Milligrams of Lactose.	Milligrams of Copper.	Milligrams of Lactose.	Milligrams of Copper.	Milligrams of Lactose.	Milligrams of Copper.	Milligrams of Lactose.
100	71.6	161	117.1	221	162.7	281	209.1	341	256.5
101	72.4	162	117.9	222	163.4	282	209.9	342	257.4
102	73.1	163	118.6	223	164.2	283	210.7	343	258.2
103	73.8	164	119.4	224	164.9	284	211.5	344	259.0
104	74.6	165	120.2	225	165.7	285	212.3	345	259.8
105	75.3	166	120.9	226	166.4	286	213.1	346	260.6
106	76.1	167	121.7	227	167.2	287	213.9	347	261.4
107	76.8	168	122.4	228	167.9	288	214.7	348	262.3
108	77.6	169	123.2	229	168.6	289	215.5	349	263.1
109	78.3	170	123.9	230	169.4	290	216.3	350	263.9
110	79.0	171	124.7	231	170.1	291	217.1	351	264.7
111	79.8	172	125.5	232	170.9	292	217.9	352	265.5
112	80.5	173	126.2	233	171.6	293	218.7	353	266.3
113	81.3	174	127.0	234	172.4	294	219.5	354	267.2
114	82.0	175	127.8	235	173.1	295	220.3	355	268.0
115	82.7	176	128.5	236	173.9	296	221.1	356	268.8
116	83.5	177	129.3	237	174.6	297	221.9	357	269.6
117	84.2	178	130.1	238	175.4	298	222.7	358	270.4
118	85.0	179	130.8	239	176.2	299	223.5	359	271.2
119	85.7	180	131.6	240	176.9	300	224.4	360	272.1
120	86.4	181	132.4	241	177.7	301	225.2	361	272.9
121	87.2	182	133.1	242	178.5	302	225.9	362	273.7
122	87.9	183	133.9	243	179.3	303	226.7	363	274.5
123	88.7	184	134.7	244	180.1	304	227.5	364	275.3
124	89.4	185	135.4	245	180.8	305	228.3	365	276.2
125	90.1	186	136.2	246	181.6	306	229.1	366	277.1
126	90.9	187	137.0	247	182.4	307	229.8	367	277.9
127	91.6	188	137.7	248	183.2	308	230.6	368	278.8
128	92.4	189	138.5	249	184.0	309	231.4	369	279.6
129	93.1	190	139.3	250	184.8	310	232.2	370	280.5
130	93.8	191	140.0	251	185.5	311	232.9	371	281.4
131	94.6	192	140.8	252	186.3	312	233.7	372	282.2
132	95.3	193	141.6	253	187.1	313	234.5	373	283.1
133	96.1	194	142.3	254	187.9	314	235.3	374	283.9
134	96.9	195	143.1	255	188.7	315	236.1	375	284.8
135	97.6	196	143.9	256	189.4	316	236.8	376	285.7
136	98.3	197	144.6	257	190.2	317	237.6	377	286.5

where T is the per cent of total solids, S the lactometer reading, and F the fat. An ingenious instrument known as Richmond's milk-scale (Fig. 41) is useful in making the calculation, instead of employing either the formula or a table. This is constructed on the principle of the slide rule, and by its use the specific gravity may be corrected to the proper temperature, and the solids calculated from the fat and specific gravity.

Babcock's formula for solids not fat is as follows:

$$\text{Solids not fat} = \left(\frac{100S - FS}{100 - 1.0753FS} - 1\right)(100 - F)2.5,$$

S being the specific gravity, and F the percentage of fat. On this formula he has prepared a table* by means of which one may calculate solids not fat agreeing quite closely with results obtained by gravimetric analysis.† The table on page 118 has been recomputed and enlarged from that of Babcock, so as to express results in total solids rather than solids not fat.

Calculation of Proteids.—Richmond‡ has devised a formula for calculating the proteids from the fat, specific gravity, total solids, and ash of milk as follows:

$$P = 2.8T + 2.5A - 3.33F - 0.7\frac{G}{D}$$

where P represents the proteids, T the total solids, A the ash, D the specific gravity, and G the "excess density" $(= 1000D - 1000)$. The proteids being thus calculated, the sugar may be computed by difference. The calculation of the proteids in this manner gives at best only a rough approximation.

Determination of Acidity.—While milk is still fresh, i.e., before it has begun to undergo lactic fermentation, it will show an acid reaction, which is sometimes expressed in terms of lactic acid. In view of the fact that

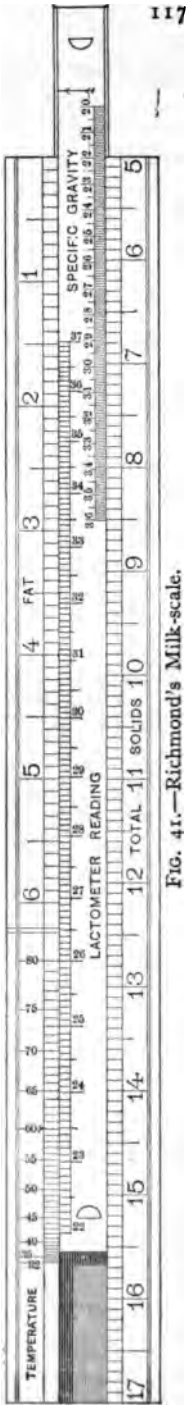


FIG. 41.—Richmond's Milk-scale.

* U. S. Dept. of Agric., Div of Chem., Bul. 47, p. 123.

† For approximate work Babcock has suggested the following simplified formulæ:

Solids not fat $= 0.25G + 0.2F$ and total solids $= 0.25G + 1.2F$, G being the lactometer reading and F the fat.

‡ Analyst, 15, p. 170.

TABLE SHOWING PER CENT OF TOTAL SOLIDS IN MILK CORRESPONDING TO QUEVENNE LACTOMETER READINGS* AND PER CENT OF FAT.†

Per Cent of Fat.	Lactometer Reading at 15.5° C.														
	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36
0.0	5.50	5.75	6.00	6.25	6.50	6.75	7.00	7.25	7.50	7.75	8.00	8.25	8.50	8.75	9.00
0.1	5.62	5.87	6.12	6.37	6.62	6.87	7.12	7.37	7.62	7.87	8.12	8.37	8.62	8.87	9.11
0.2	5.74	5.99	6.24	6.49	6.74	6.99	7.24	7.49	7.74	7.99	8.24	8.49	8.74	8.99	9.24
0.3	5.86	6.11	6.36	6.61	6.86	7.11	7.36	7.61	7.86	8.11	8.36	8.61	8.86	9.11	9.36
0.4	5.98	6.23	6.48	6.73	6.98	7.23	7.48	7.73	7.98	8.23	8.48	8.73	8.99	9.23	9.48
0.5	6.10	6.35	6.60	6.85	7.10	7.35	7.60	7.85	8.10	8.35	8.60	8.85	9.10	9.35	9.60
0.6	6.22	6.47	6.72	6.97	7.22	7.47	7.72	7.97	8.22	8.47	8.72	8.97	9.22	9.47	9.72
0.7	6.34	6.59	6.84	7.09	7.34	7.59	7.84	8.09	8.34	8.59	8.84	9.09	9.34	9.59	9.84
0.8	6.46	6.71	6.96	7.21	7.46	7.71	7.96	8.21	8.46	8.71	8.96	9.21	9.46	9.71	9.96
0.9	6.58	6.83	7.08	7.33	7.58	7.83	8.08	8.33	8.58	8.83	9.08	9.33	9.58	9.83	10.08
1.0	6.70	6.95	7.20	7.45	7.70	7.95	8.20	8.45	8.70	8.95	9.20	9.45	9.70	9.95	10.20
1.1	6.82	7.07	7.32	7.57	7.82	8.07	8.32	8.57	8.82	9.07	9.32	9.57	9.82	10.07	10.32
1.2	6.94	7.19	7.44	7.69	7.94	8.19	8.44	8.69	8.94	9.19	9.44	9.69	9.94	10.19	10.44
1.3	7.06	7.31	7.56	7.81	8.06	8.31	8.56	8.81	9.06	9.31	9.56	9.81	10.06	10.31	10.56
1.4	7.18	7.43	7.68	7.93	8.18	8.43	8.68	8.93	9.18	9.43	9.68	9.93	10.18	10.43	10.68
1.5	7.30	7.55	7.80	8.05	8.30	8.55	8.80	9.05	9.30	9.55	9.80	10.05	10.30	10.55	10.80
1.6	7.42	7.67	7.92	8.17	8.42	8.67	8.92	9.17	9.42	9.67	9.82	10.17	10.42	10.67	10.92
1.7	7.54	7.79	8.04	8.29	8.54	8.79	9.04	9.29	9.54	9.79	10.04	10.29	10.54	10.79	11.04
1.8	7.66	7.91	8.16	8.41	8.66	8.91	9.16	9.41	9.66	9.91	10.16	10.41	10.66	10.91	11.17
1.9	7.78	8.03	8.28	8.53	8.78	9.03	9.28	9.53	9.78	10.03	10.28	10.55	10.78	11.04	11.29
2.0	7.90	8.15	8.40	8.65	8.90	9.15	9.40	9.65	9.90	10.15	10.40	10.66	10.91	11.16	11.41
2.1	8.02	8.27	8.52	8.77	9.02	9.27	9.52	9.77	10.02	10.27	10.52	10.78	11.03	11.28	11.53
2.2	8.14	8.39	8.64	8.89	9.14	9.39	9.64	9.89	10.14	10.39	10.64	10.90	11.15	11.40	11.65
2.3	8.26	8.51	8.76	9.01	9.26	9.51	9.76	10.01	10.26	10.51	10.76	11.02	11.27	11.52	11.77
2.4	8.38	8.63	8.88	9.13	9.38	9.63	9.88	10.13	10.38	10.63	10.88	11.14	11.39	11.64	11.89
2.5	8.50	8.75	9.00	9.25	9.50	9.75	10.00	10.25	10.50	10.75	11.00	11.26	11.51	11.76	12.01
2.6	8.60	8.87	9.12	9.37	9.62	9.87	10.12	10.37	10.62	10.87	11.12	11.38	11.63	11.88	12.13
2.7	8.74	8.99	9.24	9.49	9.74	9.99	10.24	10.49	10.74	10.99	11.24	11.50	11.75	12.00	12.25
2.8	8.86	9.11	9.36	9.61	9.86	10.11	10.36	10.61	10.86	11.11	11.37	11.62	11.87	12.12	12.37
2.9	8.98	9.23	9.48	9.73	9.98	10.23	10.48	10.73	10.98	11.23	11.49	11.74	11.99	12.24	12.49
3.0	9.10	9.35	9.60	9.85	10.10	10.35	10.60	10.85	11.10	11.36	11.61	11.86	12.11	12.36	12.61
3.1	9.22	9.47	9.72	9.97	10.22	10.47	10.72	10.97	11.23	11.48	11.73	11.98	12.23	12.48	12.74
3.2	9.34	9.59	9.84	10.09	10.34	10.59	10.84	11.09	11.35	11.60	11.85	12.10	12.35	12.61	12.86
3.3	9.46	9.71	9.96	10.21	10.46	10.71	10.96	11.22	11.47	11.72	11.97	12.22	12.48	12.73	12.98
3.4	9.58	9.83	10.08	10.33	10.58	10.83	11.09	11.34	11.59	11.84	12.09	12.34	12.60	12.85	13.10
3.5	9.70	9.95	10.20	10.45	10.70	10.95	11.21	11.46	11.71	11.96	12.21	12.46	12.72	12.97	13.22
3.6	9.82	10.07	10.32	10.57	10.82	11.08	11.33	11.58	11.83	12.08	12.33	12.58	12.84	13.09	13.34
3.7	9.94	10.20	10.44	10.70	10.94	11.20	11.45	11.70	11.95	12.20	12.45	12.70	12.96	13.21	13.46
3.8	10.06	10.31	10.56	10.81	11.06	11.32	11.57	11.82	12.07	12.32	12.57	12.82	13.08	13.33	13.58
3.9	10.18	10.43	10.68	10.93	11.18	11.44	11.69	11.94	12.19	12.44	12.69	12.94	13.20	13.45	13.70
4.0	10.30	10.55	10.80	11.05	11.30	11.56	11.81	12.06	12.31	12.56	12.81	13.06	13.32	13.57	13.83
4.1	10.42	10.67	10.92	11.17	11.42	11.68	11.93	12.18	12.43	12.68	12.93	13.18	13.44	13.69	13.95
4.2	10.54	10.79	11.04	11.29	11.54	11.80	12.05	12.30	12.55	12.80	13.05	13.31	13.56	13.82	14.07
4.3	10.66	10.91	11.16	11.41	11.66	11.92	12.17	12.42	12.67	12.92	13.18	13.43	13.68	13.94	14.19
4.4	10.78	11.03	11.28	11.53	11.78	12.04	12.29	12.54	12.79	13.04	13.30	13.55	13.80	14.06	14.31
4.5	10.90	11.15	11.40	11.65	11.90	12.16	12.41	12.66	12.91	13.16	13.42	13.67	13.92	14.18	14.43

the acidity of "sweet" milk is due partly to the presence of acid phosphates and partly to dissolved carbonic acid in the milk, and not to lactic acid, which is probably absent, a better plan is to express the acidity in terms of the number of cubic centimeters of tenth-normal alkali necessary to neutralize a given quantity of the milk, either 25 or 50 cc., using phenolphthalein as an indicator.

If it is desired to calculate the acidity in terms of lactic acid, multiply the number of cubic centimeters of tenth-normal alkali used by 0.897, and divide by the number of cubic centimeters of milk titrated, the result being the percentage of lactic acid.

Detection of Boiled Milk.—*Dupouy's Method.*—Five cc. of milk are added to a few drops of a freshly prepared solution of diamidobenzene in water (1:4), and a little hydrogen dioxide is added. With raw milk a blue coloration will be apparent, while with milk that has been heated to 79° or over no color is produced.

MODIFIED MILK.

A comparison of the composition of cow's milk and human milk, as in the following table by Dr. Emmett Holt,* shows very marked differences.

	Woman's Milk, Average.	Cow's Milk, Average.
Fat	4.00	3.50
Sugar	7.00	4.30
Proteids	1.50	4.00
Ash	0.20	0.70
Water	87.30	87.50

The per cent of fat in the two kinds of milk is nearly the same. There is, however, too little sugar and an excess of proteids and ash in the milk of the cow, assuming human milk as the ideal infant food, so that in basing a diet for infants on the basis of human milk considerable modification is necessary. Moreover, aside from the actual variation in the amount of ingredients, there are certain inherent differences in the character of the same ingredient, as found in the milk of the cow and in human milk. The proteids of cow's milk are, for instance, found to be much more difficult of digestion than those of woman's milk, and the same is probably true of the fat. Aside from the mere statement of a

* "Infancy and Childhood."

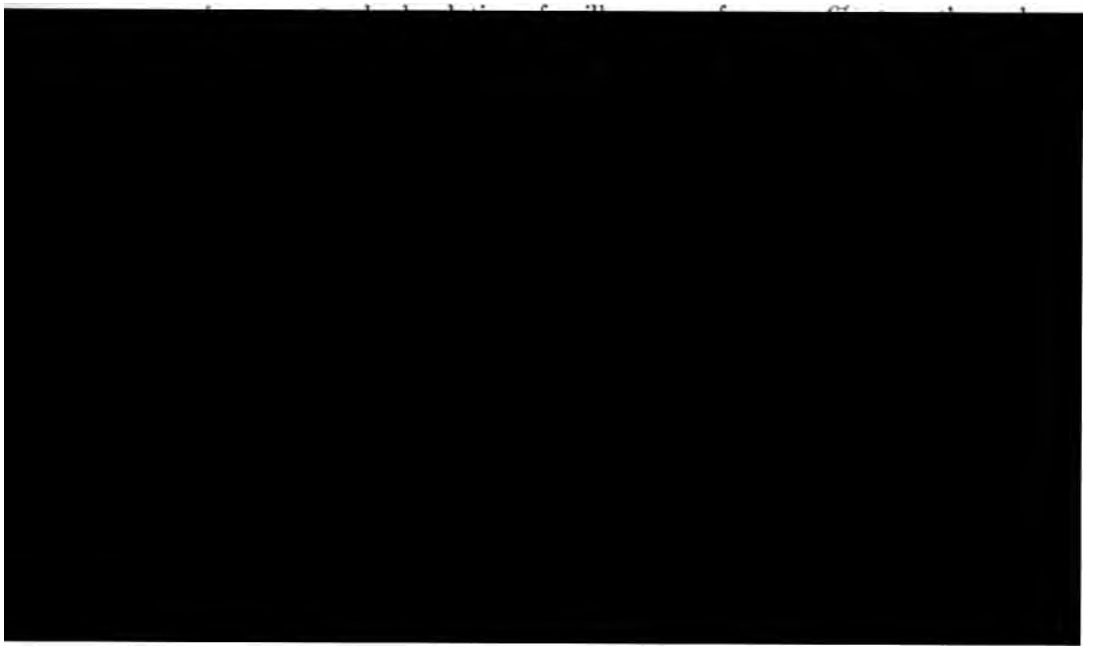
few of these differences, it is obviously beyond the scope of this work to discuss this phase of the subject in detail, reference being made, however, to such books as Dr. T. M. Rotch's "Pediatrics," and "Infancy and Childhood" by Dr. Emmett Holt, for full particulars. So great has been the demand by physicians for "modified milk" for infant feeding, that laboratories for this exclusive purpose have been established in many of the larger cities, in which not only is milk prepared in accordance with certain fixed formulæ supposed to be adapted to average infants of varying age, but milk of any desired composition is prepared, in accordance with special prescriptions of physicians to apply to individual cases.

Methods and Ingredients.—The proteids and the ash in cow's milk are much higher than in human milk, and both are brought to the proper degree of reduction by diluting the milk with water. Milk sugar is increased by the addition of lactose, and the fat is increased or diminished by addition of cream or by skimming.

The dilution of cow's milk with a measured amount of water shows the following results on the proteids and ash:

	Cow's Milk.	Diluted Once.	Diluted Twice.	Diluted Three Times.	Diluted Four Times.
	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.
Proteids.....	4.00	2.00	1.33	1.00	0.80
Ash.....	0.70	0.35	0.23	0.18	0.14

The ingredients commonly employed for modifying milk are (1) cream, containing 16% of fat, (2) centrifugally skimmed milk, otherwise known as "separator milk" from which the fat has been removed, (3) milk



Period.	Fat.	Proteids.	Sugar.
	Per cent.	Per cent.	Per cent.
Third to fourteenth day.....	2	0.6	6
Second to sixth week.....	2.5	0.8	6
Sixth to eleventh week.....	3	1.0	6
Eleventh week to fifth month..	3.5	1.5	7
Fifth to ninth month.....	4	2	7
Ninth to twelfth month.	3.5	2.5	3.5

Milk according to the above formulæ can be very simply prepared by the aid of a specially made graduate known as the "Materna" and shown in Fig. 42.

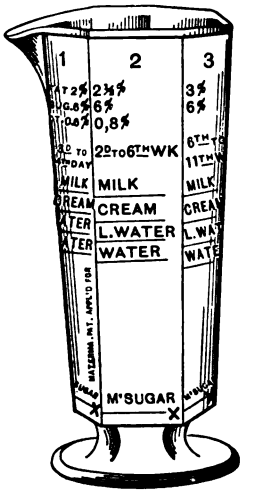


FIG. 42.—The "Materna" Graduate for Modifying Milk.

PREPARED MILK FOODS.

Milk Powder.—There are numerous brands of desiccated milk or milk powder on the market, sold in bulk and by the can, and largely used by bakers. Many of these purport to contain all the ingredients of milk excepting water, but no samples of desiccated whole milk have yet come under the notice of the writer. All the available brands examined by him have proved to be pulverized dried skimmed milk, having a composition of which the following is typical:

Moisture	8.16
Fat	1.73
Proteids (N×6.25)	33.84
Milk sugar	49.35
Ash	6.87
	99.95

This corresponds to about 0.16% fat in the original milk.

Artificial Albuminous Foods.—The albumin and casein of milk have furnished the basis of a variety of food preparations, some of which are intended for the use of invalids and people of weak digestion, and others, from their compactness, for travellers and campers. Among these foods are the following:

Nutrose This is a caseinate of sodium formed by the action of the alkali upon dried casein. It is soluble in water.

Eucasin is a caseinate of ammonium, a soluble powder somewhat similar to nutrose.

Plasmon.—This is a yellowish powder, prepared by treatment with sodium bicarbonate of the curd precipitated from skimmed milk. The compound is kneaded in an atmosphere of carbon dioxide, and reduced to a soluble powder.

The following analysis of *plasmon* was made by Woods and Merrill:*

Water.	Proteids.	Fat.	Carbohydrates.	Ash.	Fuel Value.
8.5	75.0	0.2	8.9	7.4	2044

Sanose.—This is also a powder, containing 80% of pure casein and 20% of albumose, obtained from the white of egg. The powder possesses a slight taste and an odor suggestive of milk. By briskly stirring the powder with water, an emulsion may be made much resembling milk, but on standing it soon breaks up.

Sanatogen is a grayish-white, tasteless powder, containing 95% of casein and 5% sodium glycero-phosphate. When treated with cold water it swells, forming on heating a milk-like emulsion.

Koumis is a stimulating beverage, prepared by allowing milk to undergo alcoholic, lactic, and proteolytic fermentations. The original koumis was made by the Tartar tribes of Asia from mare's milk, which contains more lactose than cow's milk, and apparently lends itself more readily to fermentation. Only a limited amount of koumis is now made from mare's milk, the milk chiefly used for this preparation being that of the cow, treated with yeast and sometimes added sugar. Koumis is a beverage much more commonly used in Europe than in America.



The proteolytic fermentation is less pronounced in kephir than in koumis. König gives the following table as the mean of twenty-eight analyses:

Water.	N tro-gen.	Casein.	Albu-min.	Acid Albu-min.	Hemi-albumin.	Pep-tone.	Fat.	Lac-tose.	Lactic Acid.	Alco-hol.	Ash.
91.21	3.49	2.53	0.36	0.21	0.21	0.039	1.44	2.41	1.02	0.75	0.68

ADULTERATION OF MILK.

Systems of Milk Inspection.—A typical method of general food inspection has already been outlined (see pp. 5 and 6), which may easily be modified to include the inspection of milk in connection with other foods, or to provide for a system of milk inspection exclusively. In the examination of such a perishable food as milk, it has not been found practicable for the analyst to reserve for the benefit of the defendant a sealed sample, as in the case of other foods, but experience has shown it had best be made the duty of the collector or inspector to give a sealed sample of milk to the dealer, when the latter requests it at the time of taking the sample. For this purpose the collector is provided with small bottles and sealing pharaphernalia, in addition to the tagged sample bottles or cans in which he collects the milk. The collector should use the same precautions for obtaining a perfectly fair representative sample as does the chemist in making the analysis, i.e., he should carefully pour the milk from the original container into an empty can or vessel and back again, before taking his sample.

Each sample is properly numbered by the collector in presence of the dealer, and the data as to the taking of the sample entered at once under the proper number in the collector's book. If a sealed sample is given, it should bear the same number as the sample reserved for analysis, and a receipt should invariably be required from the dealer, as evidence that his request for a sealed sample has been complied with.

Milk Standards Fixed by Law.—In localities where a systematic form of milk inspection prevails, there is usually in force a statute fixing the legal standard for the total solids, and in many cases for the fat or for the solids exclusive of fat. In some states the statute is so drawn that any deviation from the legal standard constitutes an adulteration in the eye of the law, and hence the offender, who has such milk in his possession with intent to sell, is liable to the same fine as if he actually added water or a foreign substance to the milk.

In other states a distinction is made by the statute between milk that is simply below the legal standard of total solids, and milk containing actually added ingredients (water or otherwise), a much lighter fine being imposed for the former than for the latter offense. Where such a distinction prevails, it often becomes incumbent upon the analyst to show to the satisfaction of the court, in case of milk low in solids, whether or not the milk has been fraudulently watered after being drawn from the cow, it being well understood that cows may give milk below the standard.

Pure milk that is low in solids may owe its deficiency either to poor feeding, or to an inherent tendency on the part of the cow to give milk always of poor quality. Thus the Holstein cow, more than any other breed, is open to the charge of sometimes giving milk below the standard.* That the Holstein cow is a favorite with the producer is by no means strange, from the fact that no other breed can with moderate feeding be made to give so large a quantity of milk.

Wherever there is a statute fixing the standard for milk, it commonly provides also that the addition of any foreign substance whatsoever constitutes an adulteration, and is not to be countenanced, in spite of the open question as to whether or not antiseptic substances, much used as preservatives, are or are not harmful.

U. S. Standards.†—*Standard milk* is milk containing not less than 12% of total solids, and not less than 8.5% of solids not fat, nor less than 3.25% of milk-fat.

Standard Skim-milk is skim-milk containing not less than 9.25% of milk solids.

FORMS OF ADULTERATION.—Milk is ordinarily adulterated (1) by watering, (2) by skimming, (3) by both watering and skimming, and (4) by the addition of one or more foreign ingredients.



termed watered, and in the other skimmed, using the terms broadly and not necessarily meaning actual and fraudulent tampering with the milk. In a third case, and almost invariably fraudulently, both watering and skimming may be found to have been practiced on the same sample. The analyst judges which of these causes have produced a milk low in solids, by a careful study of the relation between the percentages of total solids, fat, and solids not fat.

If both the total solids and solids not fat are abnormally low, and the proportion of fat to solids not fat about the same as, or higher than, in a normal milk, it is generally safe to assume that the sample has been watered; if both the total solids and the fat are well below the standard, and the solids not fat nearly normal, then the milk has undoubtedly been skimmed; if, in the third place, the total solids and the solids not fat are proportionally reduced below the standard, while the ratio of fat to solids not fat is abnormally small, it is safe to adjudge the milk to be low by reason of both skimming and watering.

Milk of Known Purity.—It is difficult to place the minimum figure for total solids, below which a milk sample may safely be pronounced by the analyst as fraudulently watered after having been drawn from the cow. Nearly nine hundred samples of milk of known purity from various breeds of cow, milked in the presence of an inspector, have been analyzed in the Department of Food and Drug Inspection of the Massachusetts State Board of Health, extending over a period of fifteen years, and among these are many samples from Holstein cows. It is extremely rare that any of these known purity samples have been found with total solids as low as 11%, though there are instances where total solids have run as low as 10%.

It is safe to assume that in the few cases on record showing less than 10.75% of total solids, either there was something decidedly abnormal about the health of the cow, or, through some accident, the cow was only partially milked, it being a well-known fact that the last fraction of the milking includes the larger percentage of fat. (See page 92.)

It is therefore nearly always safe to condemn a milk standing below 10.75 as fraudulently watered, if at the same time it has a proportionately high per cent of fat.

The average total solids of 800 samples of milk of known purity analyzed by the Massachusetts Board up to and including the year 1890 amounted to about 13¼%.

It is rare indeed to find a herd of ten or more well-fed cows of mixed

breeds in which the average milk of the herd falls below 12½% of solids.

The milk of forty-seven Holstein cows, examined in 1885, was found to contain an average of 12.51% of total solids, while the milk of eleven Jerseys examined in the same year averaged 14.02% of solids. These examples represent the two extremes commonly met with.

Variation in Standard.—In Massachusetts the law fixes a different standard for total solids in milk during the summer, or pasture-fed season, from that in force during the winter, or stall-fed period. From April to September inclusive the legal standard is 12% of total solids, of which 9% are solids not fat, and from October to March inclusive it is 13%, of which 9.3% are solids not fat. Bearing on the question of difference in normal quality of milk during the two periods, averages were taken of the milks collected by the corps of inspectors of the Massachusetts Board of Health during a month in each period, December and June being selected as most typical, and during these months all the samples were analyzed both for total solids and fat. The samples were taken from stores, milkmen, and producers, and represented as nearly as possible the milk as actually sold to the consumers. In making the averages, all samples of skimmed milk, as well as all samples standing above 17% of total solids, or under 10.75%, were deducted. The results are summarized as follows:

QUALITY OF MILK SOLD IN MASSACHUSETTS CITIES AND TOWNS IN WINTER AND SUMMER.

	December.							
	Number of Samples.	Total Solids.			Fat.			Solids not Fat. Average Per Cent.
		Highest Per Cent.	Lowest Per Cent.	Average Per Cent.	Highest Per Cent.	Lowest Per Cent.	Average Per Cent.	
Cities	497	16.86	10.88	13.21	8.59	2.40	4.37	8.74

samples examined for both months stands at just 13%, of which 4.24% is fat and 8.76 is solids not fat.

Rapid Approximate Methods of Determining the Quality of Milk.—*The Lactometer.*—A rough idea of the quality of milk can be gained by the use of the lactometer (page 95), but, in view of the fact that a low specific gravity may be the result either of a watered milk or of a milk high in fat, good judgment is necessary in connection with its use. A milk of good standard quality should have a specific gravity between the limits of 1.027 and 1.033. A watered milk would run below the former and a skimmed milk above the latter figure, though a milk unusually rich in fat would also run low. It should easily be apparent from the taste and appearance of the milk, whether a low specific gravity is due to watering or unusual richness in fat. The fact should also be recognized, that a milk sample may be far below the standard, and still show a specific gravity within the limits of pure milk, by skillfully subjecting the milk to both skimming and watering.

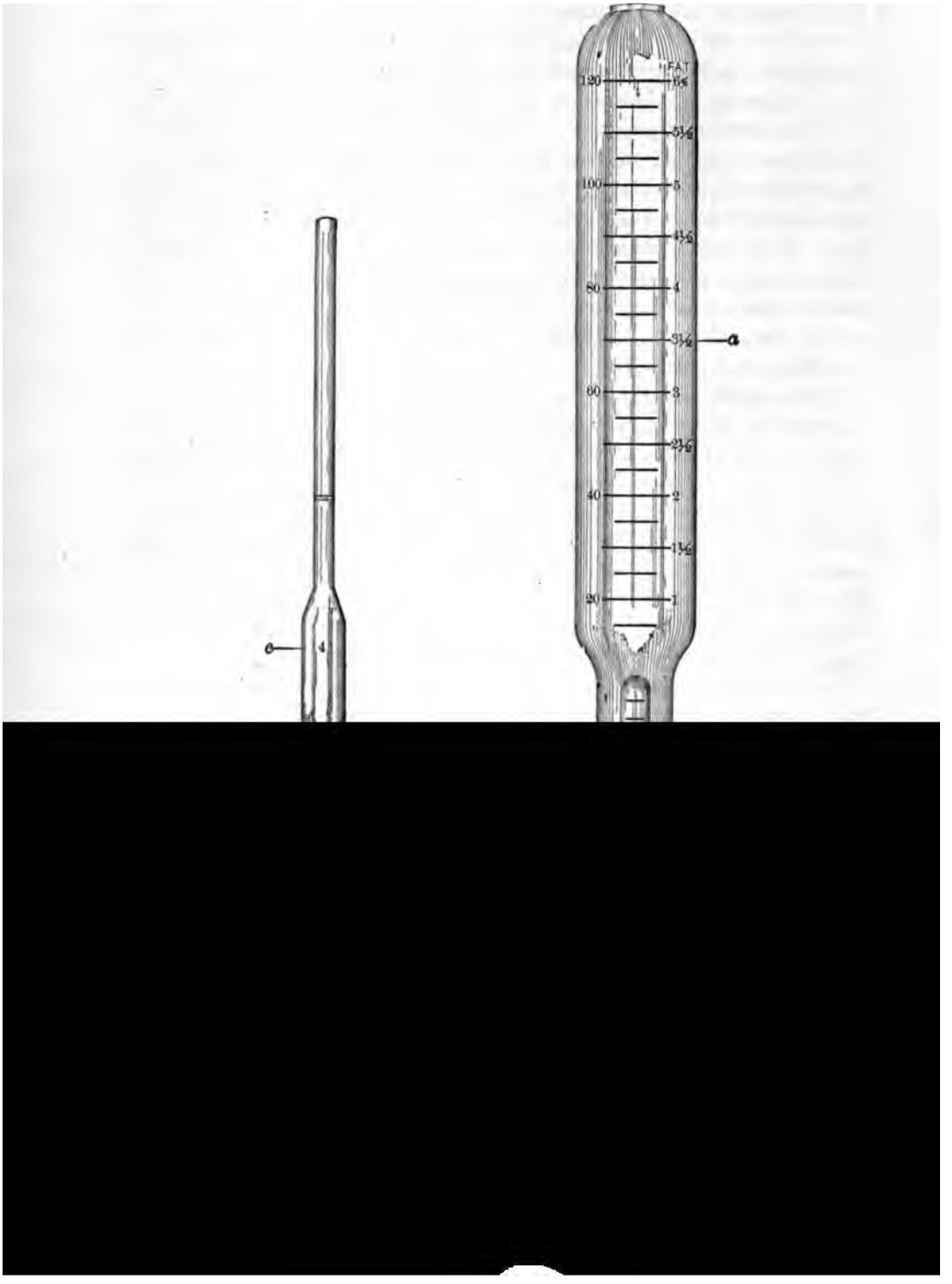
The Lactoscope.—Feser's lactoscope (Fig. 43) gives an approximation to the amount of fat in milk, and its use, especially in connection with the lactometer, is of some value. This instrument consists of a graduated glass barrel, *a*, into the bottom of which is accurately fitted the stopper, bearing a white glass cylinder, having black lines thereon. Four cc. of milk are introduced into the barrel by means of a pipette, *c*, and water is added with thorough mixing till the translucence of the mixture is sufficient to allow the black lines to be perceptible through it. The height of the level of milk and water in the barrel *a* is then read off, the number indicating roughly the percentage of fat in the sample.

As in the case of the lactometer, the purity of a milk sample cannot be positively established by the lactoscope alone. For instance, a watered milk abnormally high in fat would often be found to read within the limits of pure milk, when as a matter of fact its total solids would be below standard. By a careful comparison of the readings of both the lactoscope and lactometer, however, it is rare that a skimmed or watered sample could escape detection.

Thus, if the specific gravity by the lactometer is well within the limits of pure milk, and the fat, as shown by the lactoscope, is above 3¼ per cent., the sample may be safely passed as pure, or as conforming to the standard.

A normal lactometer reading in connection with an abnormally *low* lactoscope reading shows both watering and skimming, and with an

abnormally *high* lactoscope reading shows a milk high in fat, or a cream. With the lactoscope reading below three, and a low lactometer reading,



having in the center a shallow receptacle, the circular rim of which is raised above the level of the disk. Into this receptacle are introduced a few drops of the milk to be tested, and a circular cover-glass containing a number of variously tinted segments is placed over the receptacle, which spreads the milk out into a thin layer, and causes it to assume a tint against the black background that can be matched with one of the colors on the glass, the various tints indicating milks of various grades from the very poorest to rich cream. This test is at best a very rough one.

Examination of the Milk Serum.—*Specific Gravity.*—It has been proposed to calculate the amount of added water in milk from the specific gravity of the milk serum, since it has been found that under fixed conditions the composition of the milk serum, or clear "whey," is more constant than that of the milk itself. Hence any considerable amount of watering is manifest from the specific gravity of the serum.

In using this method the analyst should carefully work out his own standards for comparison, by personal experiment on milk of known composition to which varying amounts of water have been added, using the same conditions for obtaining the serum in all cases. Woodman's method * is as follows: To 100 cc. of the milk at a temperature of about 20° C. are added 2 cc. of 25% acetic acid, specific gravity 1.0350, in a beaker, and the beaker, covered with a watch-glass, is heated in a water-bath for 20 minutes at a temperature of 70° C. After this the beaker is placed in ice water for 10 minutes and the solution filtered. The specific gravity of the clear filtrate is taken at 15° C. with the Westphal balance.

In the Appendix will be found a table showing the specific gravity of the serum of milk containing varying degrees of added water, as well as that of the serum of a wide variety of whole milks.

Immersion Refractometer.—In the Appendix a method is given for detecting added water in milk by an examination of the serum with the Zeiss immersion refractometer.

Systematic Examination of Milk for Adulteration.—If a large number of samples of milk have to be examined daily for adulteration, it may be an advantage to submit all to a preliminary test with the lactoscope and lactometer, excluding from further analysis, as above the standard, such samples as pass certain prescribed limits which experience has proved these tests to be capable of showing to an experienced observer, and submitting the remainder to a chemical analysis. In using such an instrument as the lactoscope for this purpose, the individual element is a most

* *Jour. Am. Chem. Soc.*, 21 (1899), p. 503.

important consideration, and the use of this instrument in the milk laboratory should be limited only to a skillful operator, accustomed to interpret its results.

The method used in the writer's laboratory has been to submit all samples to the regular test for solids, and such samples as fall below the legal standard for solids, are further examined for fat.

Total Solids, Fat, and Ash.—It is presupposed that the analyst is equipped with a sufficient number of platinum dishes for the number of milk samples daily analyzed. It is a convenience to have these dishes numbered, and instead of weighing each dish, to have a system of numbered counterweights (Fig. 44) corresponding to the dishes. The counterweights in use by the author for this purpose are easily made from half-inch lead pipe, cut to the appropriate length and flattened. Each weight is then carefully adjusted to its appropriate dish, by trimming off the weight with a knife, or by adding bits of lead scraps, if necessary, by simply prying open in the center, inserting the required amount of scrap, and then closing by a blow of the hammer, the weight being plainly numbered before final adjustment. A rack is provided by the side of the balance-case (Fig. 45) with slits for holding the weights in their appropriate places. Such a set of counterweights is not difficult to make, requires very little care to keep in adjustment, and is an immense labor-saving device.



FIG. 44.—Lead Counterweight for Platinum Dish. Made from half-inch lead pipe flattened.

Details of Manipulation.—The following method of examining large numbers of milk samples is the one in use in the laboratory of the Massa-

first having thoroughly mixed the sample. This operation is repeated with all the samples, the platinum dishes containing the weighed amounts of each being placed in succession on the tray, which is finally carried to the water-bath and the dishes transferred thereto. The time required for weighing out 12 samples of milk in this manner is about fifteen minutes.

The water-bath is inclosed in a hood, and the sliding front is so arranged that it can be shut down and locked, so that if the analyst has to leave

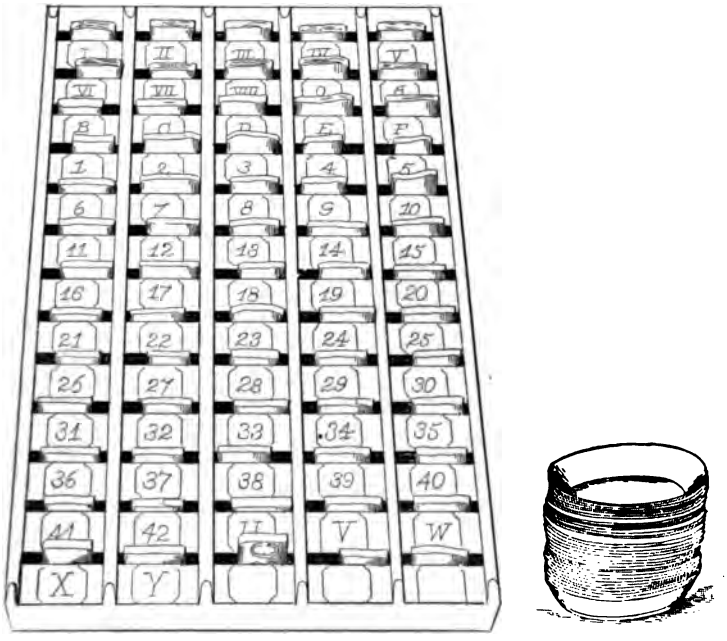


FIG. 45.—Set of Counterweights for Numbered Platinum Dishes, in a Convenient Rack.

the laboratory during the three hours required for the evaporation, he can swear in court that the samples could not be tampered with during his absence (see page 19).

When ready to make the second weighings for the total solids, each dish is taken from contact with the steam, and, while still hot, is wiped dry with a soft towel, till twelve of the dishes are placed on the tray, which is then taken to the balance. Experience has shown that with ordinary rapidity in weighing, twelve of the residues may be thus dealt with at a time without the need of a desiccator, the gathering of moisture during that time being inappreciable, excepting in very damp weather, when a less number of dishes should be removed at a time from the bath. In making the second weighing, and employing the counterweight as

Date.. January 6, 1904.

Inspector's Number.	No. of Dish	Wt. Residue 5 Grams.	Total Solids.	No. of Bottle	Fat.	Solids not Fat.	Remarks
2642 f	1	.6458	12.91				
2644	2	.6530	13.06				
2646	3	.6011	12.02				
2648	4	.5980	11.96	3	3.25	8.71	
2650	5	.7263	14.53				
2652	6	.4174	8.35	4	2.50	5.85	Colored with Annatto.
2654	7	.6823	13.65				
2656	8	.6301	12.60				
2658	9	.6924	13.85				
2660	10	.6135	12.27				
2662	11	.4595	9.19	5	0.15	9.04	Marked Skimmed.
2664	12	.4693	9.39	6	.285	6.54	
2666	13	.6530	13.06				Contains Formaldehyde
2668	14	1.1452	22.90				Contains boric acid.
2670	15	.6293	12.59				
2672	16	.7393	14.79				Sour
2674	17	.7102	14.20				
2676	18	.6010	12.02				

[redacted]

before, the exact net weight of the residue is at once ascertained and entered in the appropriate column in the note-book. Multiplied by 20 it gives at once the percentage of total solids.

It is a great saving of time to weigh out exactly 5 grams as above described. The knack of quickly measuring out the exact amount is easily acquired with practice, the 5-gram weight is the only one required for the operation with the counterweight of the dish, and the laborious figuring of percentage due to using a fraction above or below the 5 grams of milk is avoided.

Such samples as are found to stand below the standard of total solids are further examined for fat by the Babcock process (p. 100), entering the number of the fat bottle in the note-book in the appropriate column, and subsequently the percentage of fat.

Ordinarily the specific gravity is not determined, excepting in some cases of badly watered milk, when, for purposes of a check, it is customary to take the specific gravity, and calculate the solids from the gravity and the fat by Babcock's formula (p. 89), or the Richmond sliding scale, and compare the result with the figure directly determined.

The ash is rarely weighed except in special cases.

The dishes containing the dry residues are easily cleaned by first burning to an ash and cooling, after which they are treated successively with strong nitric acid, which is poured from one to another, the dishes being rinsed thoroughly with water and finally heated to redness.

A convenient device for ashing a large number of residues for purposes of cleaning the platinum dishes and for final heating is the incinerator shown in Fig. 47, made of Russia iron.

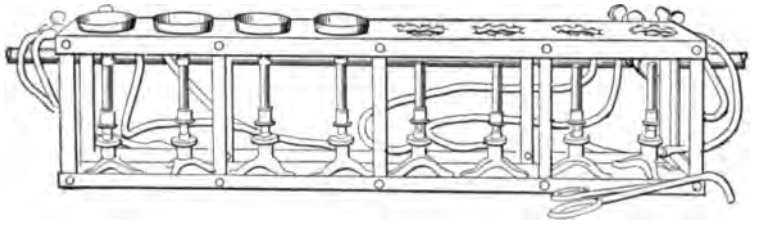


FIG. 47.—A Sheet-metal Incinerator, Specially Useful for Ashing Milk Residues.

ADDED FOREIGN INGREDIENTS.

Passing over such mythical and impossible adulterants as chalk, and the almost as rarely used substances calves' brains, starch, glycerin,

sugar, etc., often discussed in manuals on milk, but with few authentic instances of their actual occurrence, the commonly found adulterants may be divided into two classes: coloring matters and preservatives.

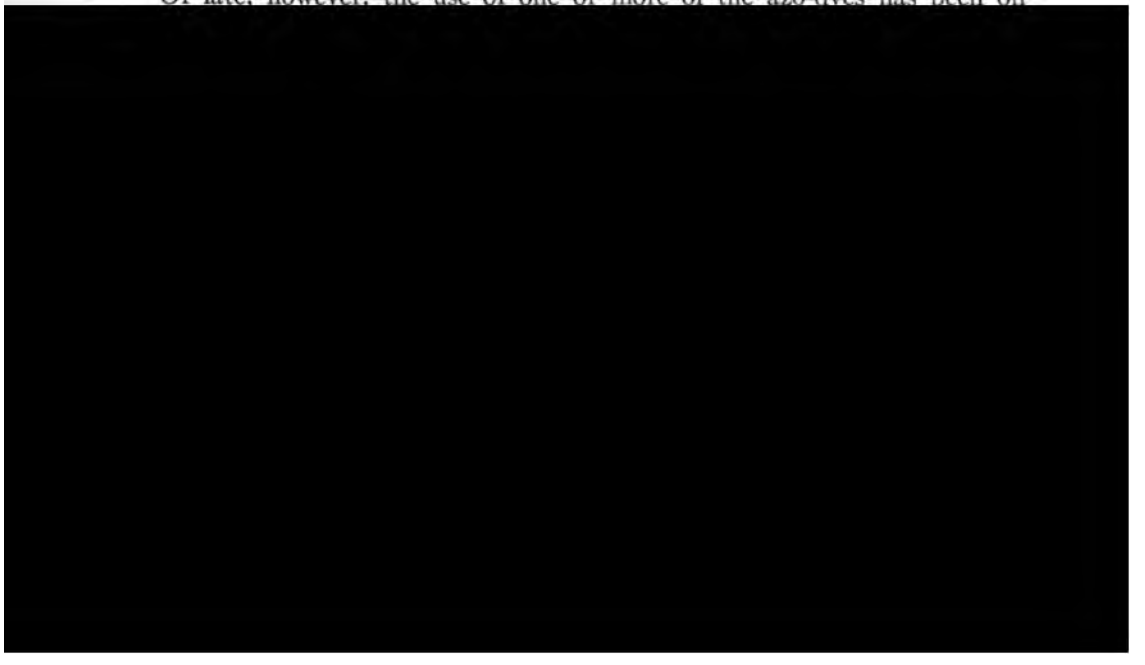
The coloring matters almost exclusively used are annatto, azo-colors, and caramel. The preservatives commonly met with are formaldehyde, boric acid, borax, and sodium bicarbonate. Rarely salicylic and benzoic acids are found.

COLORING MATTERS.—While it is more often true that an artificially colored milk is also found to be watered, the coloring being added to cover up evidence of the watering, it is not uncommon to find added coloring matter in milk above the standard.*

About 95% of the milks found colored in Massachusetts show on analysis the fraudulent addition of water.

Statistics of the Massachusetts State Board of Health show that out of 48,000 samples of milk collected throughout the state and analyzed during nine years (from 1894 to 1902 inclusive) 342 samples or 0.7% were found to contain foreign coloring matter. Of these samples, about 67% contained annatto, approximately 30% were found with an azo-dye, and about 3% with caramel.

Until comparatively recently annatto was employed almost exclusively for this purpose. Caramel is least desirable of all the above colors from the point of view of the milk-dealer, in that it is difficult to imitate with it the natural color of milk, by reason of the fact that the caramel color has too much of the brown and too little of the yellow in its composition. Annatto, on the other hand, when judiciously used and with the right dilution, gives a very rich, creamy appearance to the milk, even when watered, which accounts for its popularity as a milk adulterant. Of late, however, the use of one or more of the azo-dyes has been on



sufficient to direct the attention of the analyst to an artificially colored milk, in the course of handling a large number of samples.

Nature of Annatto.—Annatto, arnatto, or annotto is a reddish-yellow coloring matter, derived from the pulp inclosing the seeds of the *Bixa orellana*, a shrub indigenous to South America and the West Indies.

A solution of the coloring matter in weak alkali is the form usually employed in milk.

Nature of "Anilin Orange."—Of the coal-tar colors employed for coloring milk, the azo-dyes are best adapted for this purpose and are most used. A few samples of these commercial "milk improvers" have fallen into the hands of the Department of Food and Drug Inspection of the Massachusetts Board of Health, and have proved, on examination, to be mixtures of two or more members of the diazo-compounds of anilin. A mixture of what is known to the trade as "Orange G" and "Fast Yellow" gives a color which is practically identical with one of these preparations, secured from a milk-dealer and formerly used by him.

For purposes of prosecution or otherwise, it is obviously best in our present knowledge of the subject to adopt a generic name such as "a coal-tar dye" or "anilin orange"* to designate this class of coloring matters in milk, rather than to particularize.

Systematic Examination of Milk for Color.—The general scheme employed by the writer for the examination of milk samples suspected of being colored is as follows:† About 150 cc. of the milk are curdled by the aid of heat and acetic acid, preferably in a porcelain casserole over a Bunsen flame. By the aid of a stirring-rod, the curd can nearly always be gathered into one mass, which is much the easiest method of separation, the whey being simply poured off. If, however, the curd is too finely divided in the whey, the separation is effected by straining through a sieve or colander. All of the annatto, or of the coal-tar dye present in the milk treated would be found in the curd, and part of the caramel. The curd, pressed free from adhering liquid, is picked apart, if necessary, and shaken with ether in a corked flask, in which it is allowed to soak for several hours, or until the fat has been extracted, and with it the annatto. If the milk is uncolored, or has been colored with annatto, on pouring off the ether the curd should be left perfectly white. If, on

* The term "anilin orange" has been so commonly applied during the last eight years to any color or mixture of colors of this class in complaints in the Massachusetts courts, as to have acquired a special meaning perfectly well understood.

† Jour. Am. Chem. Soc., 22, 1900, p. 207.

the other hand, anilin orange or caramel has been used, after pouring off the ether the curd will be colored more or less deeply, depending on the amount of color employed. In other words, of the three colors, annatto, caramel, and anilin orange, the annatto only is extracted by ether. If caramel has been used, the curd will have a brown color at this stage; if anilin orange, the color of the curd will be a more or less bright orange.

Tests for Annatto.—The ether extract, containing the fat and the annatto, if present, is evaporated on the water-bath, the residue is made alkaline with sodium hydroxide, and poured upon a small, wet filter, which will hold back the fat, and, as the filtrate passes through, will allow the annatto, if present, to permeate the pores of the filter. On washing off the fat gently under the water-tap, all the annatto of the milk used for the test will be found to have been concentrated on the filter, giving it an orange color, tolerably permanent and varying in depth with the amount of annatto present. As a confirmatory test for annatto, stannous chloride may afterward be applied to the colored filter, producing the characteristic pink color.

Tests for Caramel.—The fat-freed curd, if colored after the ether has been poured off, is examined further for caramel or anilin orange, by placing a portion of the curd in a test-tube, and shaking vigorously with concentrated hydrochloric acid. If the color is caramel, the acid solution of the colored curd will gradually turn a deep blue on shaking, as would also the white fat-free curd of an uncolored milk, the blue coloration being formed in a very few minutes, if the fat has been thoroughly extracted from the curd; indeed, it seems to be absolutely essential for the prompt formation of the blue color in the acid solution that the curd

either wholly white, or at most of a pale straw color, while in the presence of caramel, the residue is a more or less dark-brown color, according to the amount of caramel used.

Tests for Coal-tar Dye.—If the milk has been colored with an azo-dye, the colored curd, on applying the strong hydrochloric acid in the test-tube, will immediately turn pink. If a large amount of the anilin dye has been used in the milk, the curd will sometimes show the pink coloration when hydrochloric acid is applied directly to it, before treatment with ether, but the color reaction with the fat-free curd is very delicate and unmistakable.*

Lythgoe† has shown that the amount of anilin orange ordinarily present in a milk for the purposes of coloring can be detected by adding directly to say 10 cc. of the sample an equal quantity of strong hydrochloric acid and mixing, whereupon the pink coloration is produced, if the dye is present in more than minute traces. The test is more delicate if carried out in a white porcelain dish. It had best be used as a preliminary test only, and confirmed by a subsequent test on the fat-free curd as above.

SUMMARY OF SCHEME FOR COLOR ANALYSIS.

Curdle 150 cc. milk in casserole with heat and acetic acid. Gather curd in one mass. Pour off whey, or strain, if curd is finely divided. Macerate curd with ether in corked flask. Pour off ether.

Ether Extract.	**Extracted Curd.**	
Evaporate off ether, treat residue with NaOH and pour on wetted filter. After the solution has passed through, wash off fat and dry filter, which if colored orange, indicates presence of annatto. (Confirm by $SnCl_2$.)	(1) *If Colorless.*—Indicates presence of no foreign color other than in ether extract. (2) *If Orange or Brownish.*—Indicates presence of anilin orange or caramel. Shake curd in test-tube with concentrated hydrochloric acid.	
	If solution *gradually* turns blue, indicative of caramel. (Confirm by testing for caramel in whey of original milk.)	If orange curd *immediately* turns pink, indicative of anilin orange.

NATURE OF PRESERVATIVES USED IN MILK.—In most localities having pure food laws preservatives in milk are regarded as adulterants.

* Occasional samples of milk colored with a coal-tar dye of a different class from those already described have recently been found in Massachusetts. In these cases the color of the separated fat-free curd does not change when treated with hydrochloric acid. The color of the curd is, however, very marked, being deep orange, bordering on the pink.

† Jour. Am. Chem. Soc., 22, 1900, p. 813.

Their use, however, seems to be on the increase. Of 6,186 samples of milk examined by the Massachusetts State Board of Health during one year (1899) 71 samples, or 1.2%, were found to contain a preservative. Of these 55 were found with formaldehyde, 13 containing boric acid, borax, or a mixture of the two, and 3 contained carbonate of soda.

Comparative tests have been made in the writer's laboratory of the keeping qualities of these commonly used milk preservatives, when present in varying strength, the milk being kept during the experiment at the temperature of the room, which at that season of the year (February) was about 20° C.* The preservatives were added about five hours after milking. The samples were titrated for acidity each morning, the acidity being expressed by the number of cubic centimeters of decinormal sodium hydroxide necessary to neutralize 5 cc. of the milk.

The proportions of preservatives used in this experiment, as shown in the table on page 139, were intended to cover a wide range, from the weakest that could aid in preserving the milk up to a strength limited only by being perceptible to the taste. The table opposite shows the results.

Formaldehyde, the most commonly used preservative for milk, is sold to the trade under various names, such as "Preservaline" "Freezine," "Iceline," etc., all being dilute aqueous solutions of formaldehyde, containing from 2 to 6 per cent of the gas, being nearly always diluted from the 40% solution known as formalin. These preparations are usually accompanied by directions, which specify the amount to be used, varying from a tablespoonful of the solution in 5 to 10 gallons of the milk. It is commonly used in the strength of 1 part of the gas in 20,000, and rarely less than 1 part in 50,000. The antiseptic power of formaldehyde increases in a marked degree as the strength of the preservative is increased. Milk

ACTION OF VARIOUS PRESERVATIVES ON MILK.

Preservative.	First Day.		Second Day.		Third Day.			Fourth Day.			Fifth Day.		
	Acidity.	Condition.	Acidity.	Condition.	Acidity.	Condition.	When Curdled.	Acidity.	Condition.	When Curdled.	Acidity.	Condition.	When Curdled.
Blank	0.78	Sweet	0.96	Sweet	3.95	Sour	1½ days	—	—	—	—	—	—
Sodium carbonate:													
1 : 1000	—	Sweet	0.75	Sweet	4.05	Sour	1½ days	—	—	—	—	—	—
1 : 500	—	"	0.75	"	4.05	"	1½ days	—	—	—	—	—	—
1 : 250	—	"	0.75	"	4.05	"	2 days	—	—	—	—	—	—
Boric acid 75%, borax 25%:													
1 : 2000	—	Sweet	0.96	Sweet	4.04	Sour	1½ days	—	—	—	—	—	—
1 : 1500	—	"	1.05	"	1.64	Sweet	—	4.28	Sour	2½ days	—	—	—
1 : 1000	—	"	1.19	"	1.44	"	—	4.31	"	3 days	—	—	—
1 : 500	—	"	1.50	"	1.52	"	—	2.18	Sweet	—	3.80	Sour	4 days
Boric acid 25%, borax 75%:													
1 : 2000	—	Sweet	1.00	Sweet	2.77	Sweet	—	4.18	Sour	2½ days	—	—	—
1 : 1500	—	"	1.00	"	1.95	"	—	4.21	"	"	—	—	—
1 : 1000	—	"	1.04	"	1.27	"	—	4.25	"	3 days	—	—	—
1 : 500	—	"	1.12	"	1.40	"	—	1.71	Sweet	—	4.20	Sour	4 days
Formaldehyde:													
1 : 50000	0.78	Sweet	0.80	Sweet	1.09	Sweet	—	3.92	Sour	2¾ days	—	—	—
1 : 20000	0.78	"	0.79	"	0.81	"	—	1.26	Sweet	—	4.15	Sour	4 days
1 : 10000	0.78	"	0.78	"	0.80	"	—	0.91	"	—	0.96	Sweet	—
1 : 5000	0.78	"	0.78	"	0.79	"	—	0.87	"	—	0.89	"	—

Preservative.	Sixth Day.		Seventh Day.			Eighth Day.		Ninth Day.		Tenth Day.		Eleventh Day.		
	Acidity.	Condition.	Acidity.	Condition.	When Curdled.	Acidity.	Condition.	Acidity.	Condition.	Acidity.	Condition.	Acidity.	Condition.	When Curdled.
Blank	—	—	—	—	—	—	—	—	—	—	—	—	—	—
Sodium carbonate:														
1 : 1000	—	—	—	—	—	—	—	—	—	—	—	—	—	—
1 : 500	—	—	—	—	—	—	—	—	—	—	—	—	—	—
1 : 250	—	—	—	—	—	—	—	—	—	—	—	—	—	—
Boric acid 75%, borax 25%:														
1 : 2000	—	—	—	—	—	—	—	—	—	—	—	—	—	—
1 : 1500	—	—	—	—	—	—	—	—	—	—	—	—	—	—
1 : 1000	—	—	—	—	—	—	—	—	—	—	—	—	—	—
1 : 500	—	—	—	—	—	—	—	—	—	—	—	—	—	—
Boric acid 25%, borax 75%:														
1 : 2000	—	—	—	—	—	—	—	—	—	—	—	—	—	—
1 : 1500	—	—	—	—	—	—	—	—	—	—	—	—	—	—
1 : 1000	—	—	—	—	—	—	—	—	—	—	—	—	—	—
1 : 500	—	—	—	—	—	—	—	—	—	—	—	—	—	—
Formaldehyde:														
1 : 50000	—	—	—	—	—	—	—	—	—	—	—	—	—	—
1 : 20000	—	—	—	—	—	—	—	—	—	—	—	—	—	—
1 : 10000	1.29	Sweet	4.10	Sour	5½ days	—	—	—	—	—	—	—	—	—
1 : 5000	0.92	"	0.93	Sweet	—	0.94	Sweet	0.96	Sweet	1.10	Sweet	1.32	Sour	10½ days

Whether or not formaldehyde in milk is harmful to processes of digestion, when present in the amount commonly used, is still an open question.*

Carbonate and Bicarbonate of Soda.—These substances are occasionally used in milk, though, as the above table shows, they possess little or no value as milk preservatives. They do, however, serve to neutralize the acidity of slightly soured milk and to postpone the time of actual curdling.

Salicylic and Benzoic Acids, in view of the much more efficient antiseptics at hand, are now rarely used as milk preservatives, though the analyst should be on the outlook for them. Salicylic acid is a poor milk preservative, in view of the fact that it affects the taste of the milk, when present in sufficient quantity, to be of service.

Detection of Formaldehyde.—*Hydrochloric Acid Test.*†—Commercial hydrochloric acid (specific gravity 1.2) containing 2 cc. of 10% ferric chloride per liter is used as a reagent. Add 10 cc. of the acid reagent to an equal volume of milk in a porcelain casserole, and heat slowly over the free flame nearly to boiling, holding the casserole by the handle, and giving it a rotary motion while heating to break up the curd. The presence of formaldehyde is indicated by a violet coloration, varying in depth with the amount present. In the absence of formaldehyde, the solution slowly turns brown. By this test 1 part of formaldehyde in 250,000 parts of milk is readily detected before the milk sours. After souring, the limit of delicacy proves to be about 1 part in 50,000.

Various aldehydes, when introduced into milk, give color reactions under the above treatment, but formaldehyde alone gives the violet coloration, which is perfectly distinguishable and unmistakable.

Hehner's Sulphuric Acid Test.—To 5 to 10 cc. of milk in a wide test-



the junction of the two liquids indicates formaldehyde. This test is not as delicate as the hydrochloric acid test, by reason of the charring action of the sulphuric acid.

Confirmatory Tests with Distilled Milk.—If it is desired to confirm the above tests by further evidence, 100 to 200 cc. of the milk sample are subjected to distillation, and the first 20 cc. of the distillate are used for testing.

(1) To a few drops of this distillate in a test-tube add a drop of Schiff's reagent.* In presence of any aldehyde, a pink coloration will soon be perceptible, deepening in intensity on standing.

(2) Add to 5 cc. of the milk distillate a few drops of a 1% aqueous solution of resorcin or phenol, and proceed as directed on page 666 (preservatives). The crimson color indicates formaldehyde, and not other aldehydes.

(3) Use 1 or 2 cc. of the milk distillate and apply the phenylhydrazine test, page 666.

(4) A small amount of the distillate from milk (which prior to distilling is acidified slightly with sulphuric acid to fix any free ammonia) is treated with a few drops of Nessler's reagent.† Traces of formaldehyde produce a yellow coloration, while if considerable formaldehyde be present, the color darkens on standing and a grayish precipitate may be formed.

Determination of Formaldehyde in Milk.‡—To 100 cc. of milk add 1 cc. of 1:3 sulphuric acid and subject to distillation in a 500-cc. Kjeldahl nitrogen-flask, using a low circular evaporating burner to avoid frothing. According to Smith, the first 20 cc. of the distillate, or one-fifth the original volume, contain very nearly one-third of the total formaldehyde. Collect 20 cc. of the distillate and determine the formaldehyde therein by the potassium cyanide method, as follows:§

Treat 10 cc. of tenth-normal silver nitrate with 6 drops of 50% nitric acid in a 50-cc. flask, add 10 cc. of a solution of potassium cyanide containing 3.1 grams of KCN in 500 cc. of water, and make up to the 50-cc. mark. Shake, filter, and titrate 25 cc. of the filtrate with tenth-normal ammonium sulphocyanate,‖ using ferric chloride as an indicator.

* Table of reagents, No. 226.

† Table of reagents, No. 187.

‡ Smith, Jour. Am. Chem. Soc., 25, 1903, pp. 1032 and 1037.

§ Zeits. anal. Chem., 36, pp. 18–24.

‖ Theoretically 7.6 grams per liter. On account of the deliquescent nature of the salt weigh out 8 grams, make up to a liter, and titrate against tenth-normal silver nitrate for its exact value, using ferric chloride as an indicator. Sutton, Volumetric Analysis, 8th Ed. p. 155.

Acidify another portion of 10 cc. of tenth-normal silver nitrate with nitric acid, add 10 cc. of the potassium cyanide solution to which the above 20 cc. of the formaldehyde distillate has been added. Make up the whole to 50 cc., filter and titrate as before 25 cc. of the filtrate with tenth-normal ammonium sulphocyanate for the excess of silver.

The amount of potassium cyanide used up by the formaldehyde, in terms of tenth-normal ammonium sulphocyanate, is found by multiplying by two the difference between the two results, and the total formaldehyde is calculated by multiplying by 3 the amount found in the 20 cc. of distillate.

The reaction that takes place between the formaldehyde and the potassium cyanide probably results in the formation of an addition product as follows:

$$CH_2O + KCN = KO.CH_2CN.$$

Detection of Boric Acid.—This is best accomplished by the turmeric-paper test in a solution of the ash of the milk, slightly acidified by hydrochloric acid, in the manner described on page 669.

Determination of Boric Acid.—Use the method of Thompson.* Add 10 cc. of a 1 : 1 solution of sodium hydroxide to 100 cc. of the milk, evaporate to dryness in a platinum dish, and proceed as described on page 669.

Detection of Carbonate and Bicarbonate of Soda. — The addition of carbonates is manifest by the effervescence caused by treating the milk-ash with acid. Effervescence in the milk-ash is quite perceptible, when as much as 0.05% of sodium carbonate is present.

Schmidt's method of detecting sodium carbonate or bicarbonate, when present to the extent of 0.1% or more, is as follows: Ten cc. of milk are mixed with an equal volume of alcohol and a few drops of a

tion of an emulsion. A volume of ether largely in excess over that of the curdled milk has been found to be less apt to emulsionize.* Transfer the ether extract to a separatory funnel, and separate the benzoic acid from the fat by shaking out with dilute ammonia, which takes out the former as ammonium benzoate. Evaporate the ammonia solution in a dish over the water-bath till all free ammonia has disappeared, but before getting to dryness, add a few drops of ferric chloride reagent.

The characteristic flesh-colored precipitate indicates benzoic acid. Care should be taken not to add the ferric chloride till all the ammonia has been driven off, otherwise a precipitate of ferric hydrate is formed.

Detection of Salicylic Acid.—(1) To 50 cc. of the milk add 1 cc. of acid nitrate of mercury reagent (p. 111), shake and filter. The filtrate, which should be perfectly clear, is then shaken with ether in a separatory funnel, the ether extract evaporated to dryness, and a drop of ferric chloride reagent applied. If salicylic acid be present, a violet color will be produced. In carrying out the test it should be noted that a small portion only of the salicylic acid is in the filtered whey, the larger part being left in the curd. The color test is, however, so delicate as to show its presence, when an appreciable amount is used.

(2) Proceed exactly as directed for benzoic acid (p. 142). On applying the ferric chloride to the final solution, after evaporation of the ammonia, a violet color shows the presence of salicylic acid.

Routine Inspection of Milk for Preservatives.—It is customary in the writer's laboratory to examine all the samples of milk collected during the months of June, July, August, and September for the commonly used preservatives, in addition to the regular analysis for total solids and fat. The number of samples thus examined amounts to upwards of 500 per month, varying from 10 to 60 per day. The results of such an examination during four years are thus shown:†

PRESERVATIVES IN MILK.

Year.	Samples Examined.	Number containing Formaldehyde.	Per Cent containing Formaldehyde.	Number containing Boric Acid.	Per Cent containing Boric Acid.	Number containing Carbonate.	Total containing Preservative.
1898.....	1046	26	2.5	11	1.0	4	41
1899.....	2105	55	2.6	13	0.6	3	71
1900.....	2018	61	3.0	6	0.3	–	67
1901.....	2154	42	1.9	12	0.5	–	54
1902.....	1934	29	1.5	14	0.7	–	43
Totals....	9257	213	2.3	56	0.6	7	376

* When this process is used the ether may readily be recovered by distillation.

† An. Rep. Mass. State Board of Health, 1902, p. 474; Analyst's Reprint, p. 22.

Such a system by no means involves a large amount of time or labor, and is really essential before passing judgment upon the purity of the milk, since, unlike added color, there is nothing in the physical appearance of the milk to suggest the presence of preservatives, nor are they rendered apparent by the taste, if skilfully used.

The methods employed are carried out as follows:*

(1) *Formaldehyde.*—After having been examined for total solids and fat, the milk samples are arranged in order in their original containers, and about 10 cc. of each sample are poured into a casserole and tested in succession by means of the hydrochloric acid and ferric chloride test (p. 140). A large stock bottle, which may be fitted with a siphon if desired, is kept on hand containing the hydrochloric acid reagent. Less than one minute is required in making the formaldehyde test for each sample.

2. *Carbonate and Boric Acid.*—These tests have been so simplified as to be, as it were, a side issue in the process of cleaning the platinum dishes used for the determination of total solids. The various residues from the total solids are burnt to an ash in the original numbered dishes in succession, these dishes, after incineration, being arranged side by side on a flat tray. By means of a pipette, one or two drops of dilute hydrochloric acid are introduced into each dish in succession, noting at the time any effervescence that may ensue, which is in itself an indication of sodium carbonate. After every milk ash has been acidulated, a few cubic centimeters of water are added to each dish by means of a wash-bottle, the dissolving of the ash being hastened by giving a rotary motion to the tray containing the dishes. A strip of turmeric-paper is then allowed to soak for a minute or so in each dish, after which it is withdrawn from contact with the solution and allowed to adhere to the side of the dish



with numbers marked on the paper at close intervals corresponding to the numbers of the platinum dishes. The strips of turmeric-paper, after soaking, are removed from the dishes and placed across the glass tubes, over the numbers corresponding to those of the dishes from which they were taken. Here they are allowed to stand till dry, being kept in position by a third section of tube or rod placed over them. When dry, the

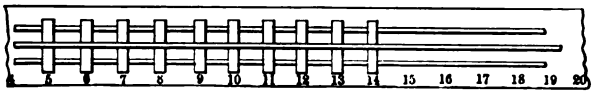


FIG. 48.—Arrangement of Glass Rods and Turmeric Test-papers in Detection of Boric Acid in Milk.

color of the turmeric strips will indicate whether or not boric acid is present, and also the position will show in what sample to look for it.

Cane Sugar.—This is alleged to be added for the purpose of increasing the total solids of milk, but if present to any marked degree, it could hardly fail of detection by reason of the sweet taste imparted to the milk. Cane sugar in milk may be detected * by boiling 5 to 10 cc. of the sample with about 0.1 gram of resorcin and a few drops of hydrochloric acid for a few minutes. In the presence of cane sugar, a rose-red color is produced.

According to Richmond, cane sugar may be estimated by first ascertaining the total polarization of the sample as in the estimation of milk sugar (p. 112). The milk sugar is then determined by Fehling's solution (pp. 113 to 115) either volumetrically or gravimetrically. The difference between the anhydrous milk sugar found by the latter, or Fehling method, and that calculated by dividing the polarization by 1.217 will give the percentage of cane sugar present.

Cotton's † method of detecting cane sugar, when present to the extent of 0.1%, consists in mixing in a test-tube 10 cc. of the suspected milk with 0.5 gram of powdered ammonium molybdate. Ten cc. of milk of known purity, or 10 cc. of a 6% solution of milk sugar are similarly treated by way of comparison. Both tubes are placed in a water-bath and the temperature gradually raised to 80° C. If cane sugar is present, an intense blue coloration is produced, while the genuine milk or the solution of milk sugar remains unchanged at the temperature of 80°. If the temperature is raised to the boiling-point, however, the pure milk or milk sugar solution may also turn blue.

* Richards and Woodman, Air, Water, and Food, p. 166

† Abs. Analyst, 1898, p. 37.

Detection of Starch in Milk.—A small quantity of milk is heated in a test-tube to boiling, cooled, and a drop of iodine in potassium iodide added. A blue coloration indicates starch.

Condensed Skimmed Milk as an Adulterant.—The use of condensed unsweetened skimmed milk to raise the solids of a skimmed or watered milk above the standard has been noted in Massachusetts. This sophistication is rendered apparent by the abnormally high solids not fat of the sample, which in some instances have exceeded 11%. A solid not fat in excess of 10% is suspicious of this form of adulteration. By fixing a legal standard for both fat and solids not fat, such tampering with milk may readily be checked.

Analysis of Sour Milk.—It occasionally becomes necessary for the analyst to deal with samples of sour milk, especially in the summer-time, when the milk has been brought from a long distance. While the process of lactic fermentation results in the formation of traces of volatile acids, unless the sample has become so badly curdled as to render an even homogeneous mixture of the various parts impossible, a fair determination of the solids and fat can readily be made. Experience has proved that, excepting in instances of milk so badly soured as to have become actually putrid, the analysis of sour milk, if carefully made, should not differ materially from that of the same milk before souring.

Care must be taken to secure an even emulsion of the curd and whey. This may sometimes be accomplished by repeatedly pouring the sample back and forth from one container to another. Again, it is sometimes necessary to use an egg-beater of the spiral wire pattern, which preferably should easily fit the can or milk-container. Unless a fine, even emulsion can be secured, it is impossible to make a satisfactory analysis of sour milk. With such an emulsion results can be relied on



as found on the market usually nothing better than condensed ordinary milk, having no added sugar, and has generally no resemblance in composition to cream other than in consistency.

Condensed milk is usually prepared by boiling milk in vacuum-pans under diminished pressure to the proper degree of concentration. Upwards of 350 samples of sweetened condensed milk have been analyzed in full in the laboratory of the Massachusetts State Board of Health during the last eight years, representing no less than 110 brands, together with about 30 samples (representing 8 brands) of the unsweetened variety.

In view of the fact that a considerable number of the condensed-milk samples are shown by their analysis to have been produced from skimmed milk, the fat content in the samples analyzed varying from a mere trace to 12%, it is obvious that the typical composition of condensed milk could not fairly be shown by giving maximum, minimum, and mean results from the entire tabulated series, nor would it be possible to draw a hard-and-fast line excluding certain samples known to be adulterated in making up the averages. It has therefore been thought best to select a few typical brands and give their analyses in full.

COMPOSITION OF SWEETENED CONDENSED MILK.

Points to be Noted.	Total Solids, Per Cent.	Water Per Cent.	Milk Solids, Per Cent.	Cane Sugar, Per Cent.	Milk Sugar, Per Cent.	Proteids, Per Cent.	Fat, Per Cent.	Ash, Per Cent.	Fat in Original Milk, Per Cent.
High in fat, much added sugar.	79.17	20.83	31.32	47.85	9.57	7.95	12.00	1.80	4.60
High fat, low milk sugar.	68.70	32.30	30.27	38.43	6.38	10.70	11.46	1.73	5.63
Low fat, high milk sugar; low proteids.	69.30	30.70	31.83	37.47	16.75	6.34	7.20	1.54	2.77
Normal constituents throughout*.	74.29	25.71	32.37	41.92	11.97	8.46	10.65	1.29	4.6
Condensed from skimmed milk.	69.30	30.70	29.15	40.15	11.89	12.15	3.06	2.05	1.11
Condensed from centrifugally skimmed milk.	69.06	30.94	25.88	43.18	11.55	11.78	0.09	2.46	Trace

COMPOSITION OF UNSWEETENED CONDENSED MILK.

Points to be Noted.	Total Solids, Per Cent.	Water, Per Cent.	Milk Sugar, Per Cent.	Proteids, Per Cent.	Fat, Per Cent.	Ash, Per Cent.	Fat in Original Milk, Per Cent.	No. of Times Condensed.
High in fat.	36.00	64.00	10.65	11.63	12.00	1.72	4.61	2.6
Low in proteids.	31.25	86.75	13.40	7.02	9.60	1.23	4.18	2.3
Normal constituents throughout*	28.16	69.24	9.85	8.66	8.10	1.55	3.68	2.2
Condensed from skimmed milk.	35.17	64.83	13.90	15.37	4.20	1.70	1.28	3.3

* Can be taken as being very near the average for all constituents in honest condensed milk of fair quality.

In the case of sweetened condensed milk it will be observed that the proteids as a rule run considerably lower than the sugar, whereas in ordinary cow's milk the percentage of proteids and milk sugar are more nearly alike. In making the above analyses all the reducing sugar was reckoned as milk sugar, whereas it is possible that a small amount of the cane sugar is inverted in the process of manufacture, and thus increases the amount of reducing sugar.

U. S. Standards.*—*Standard condensed milk* and *standard sweetened condensed milk* are condensed milk and sweetened condensed milk respectively, containing not less than 28% of milk solids, of which not less than one-fourth is milk fat. Standard condensed skim-milk, is skim-milk from which a considerable portion of water has been evaporated.

Methods of Analysis.—*Preparation of the Sample.*—For the analysis of condensed milk the following system of procedure has been adopted in the laboratory of the Massachusetts State Board of Health. The sample is first thoroughly mixed, best by transferring the entire contents of the can to a large evaporating-dish, and working it thoroughly with a pestle till homogeneous throughout. Forty grams of the mixed sample are weighed out, preferably in a tared weighing-tray for sugar analysis, transferred by washing to a graduated 100-cc. sugar-flask (or if desired it is weighed directly into the flask) and made up to the mark with water.

Total Solids.—An aliquot part of this mixed solution is further diluted with an equal amount of water, and 5 cc. of the diluted mixture, corresponding to 1 gram of the condensed milk, is pipetted into a tared platinum dish, such as is used for ordinary milk, the pipette being rinsed into the



skimmed milk, be caked down hard and glossy on the bottom of the dish, but, if the operation is properly carried out, should have a well-separated fat layer at the top, and the residue should resemble in appearance that from ordinary milk. This result is accomplished by the extreme dilution of the sample.

Ash.—The residue from the total solids as above obtained is carefully burnt, cooled, and weighed as in the case of ordinary milk (p. 98).

When the total solids are not to be determined, as in cases where the quality of the milk used in preparation of the sample is decided by the fat and ash alone (see p. 152), 12.5 cc. of the above 40% solution, corresponding to 5 grams of the sample, are evaporated to dryness on the water-bath, and the residue burnt to an ash in the muffle or over a low flame.

*Fat.—The Author's Method.**—Fifteen cc. of the 40% solution prepared as above described, corresponding to 6 grams of the original condensed milk, are measured into an ordinary test-bottle of the Babcock centrifuge. This is filled nearly to the neck with water, and 4 cc. of a solution of copper sulphate of the strength of Fehling's copper solution are added. The contents are thoroughly shaken, and the precipitated proteids, carrying with them the fat are rapidly separated out by whirling the fat bottle in the centrifuge, preferably without heating. The writer prefers an electric centrifuge of the Robinson type (p. 101) for this purpose, as the heat of the steam-driven machine cakes the precipitate down, so that it is harder to wash. If desired, the precipitate may be allowed to settle out of itself, which it does more quickly in the cold.

The supernatant liquid containing the sugar is drawn off by means of a pipette of large capacity, having a stem sufficiently small to pass easily into the neck of the milk-bottle, a small wisp of absorbent cotton being first twisted over the bottom of the pipette to serve as a filter. On withdrawing the pipette with the sugar solution, the cotton is wiped off into the bottle by rubbing against the inner side.

The precipitated proteids and fat are given two additional washings, as above, by shaking thoroughly with water introduced nearly to the neck of the bottle, separating out in each case by centrifuge or by settling, and finally removing the washings with the pipette, two of such extra washings being found nearly always sufficient to remove all the sugar. If the precipitate is caked down hard after treatment with the centrifuge,

* 28th An. Rep. Mass. State Board of Health, 1896, p 630, and Jour. Am. Chem. Soc., 22, 1900, p. 589.

it may be necessary to employ a stiff platinum wire as a stirrer to aid in mixing with the wash-water.

Finally, enough water is added to amount approximately to the normal volume of 17.6 cc. usually employed for the Babcock test, 17.5 cc. of sulphuric acid are added, and the test continued from this point on as in the ordinary Babcock process of milk-testing, multiplying the reading obtained by three to give the correct percentage of fat in the sample.

For condensed milk containing no added cane sugar, these precautions are, of course, unnecessary, the ordinary Babcock method being directly employed with a weighed portion of the milk.

Proteids.—Five cc. of the 40% solution originally prepared, corresponding to 2 grams of the condensed milk, are diluted further to about 40 cc., and just enough of the Fehling copper solution is added, drop by drop, to precipitate the albuminoids, taking care to avoid a large excess. As a rule, 0.6 cc. of copper solution is ample for this. Nearly neutralize with sodium hydroxide, stopping just short of alkalinity, i.e., leaving the solution still *slightly acid.* An excess of alkali tends to dissolve the casein and cause turbidity in the filtrate. Pass through a weighed filter-paper, wash, dry in an air-oven at 100° C., and weigh. The filter with the dry precipitate is then carefully burnt in a porcelain crucible, and the difference between the weight of the dry precipitate and the weight of the ash is the weight of the proteids and fat. Expressing this in percentage, and deducting from it the per cent of fat previously obtained, the result is the per cent of proteids.

Milk Sugar.—The filtrate and the washings from the preceding operation are made up to 100 cc. with water, and the amount of reducing sugar obtained volumetrically by Fehling's solution is reckoned as

PER CENT MILK SUGAR CORRESPONDING TO NUMBER OF CUBIC CENTIMETERS USED.

Strength of solution 2 grams in 100 cc.

Cu. Cm.	Per Cent.	Cu. Cm.	Per Cent.	Cu. Cm.	Per Cent.	Cu. Cm.	Per Cent.
18.0	18.61	25.0	13.40	32.0	10.47	39.0	8.59
18.5	18.10	25.5	13.14	32.5	10.31	39.5	8.49
19.0	17.63	26.0	12.89	33.0	10.15	40.0	8.37
19.5	17.18	26.5	12.64	33.5	10.00	40.5	8.27
20.0	16.75	27.0	12.41	34.0	9.85	41.0	8.17
20.5	16.34	27.5	12.18	34.5	9.71	41.5	8.07
21.0	15.95	28.0	11.97	35.0	9.57	42.0	7.98
21.5	15.58	28.5	11.75	35.5	9.43	42.5	7.88
22.0	15.22	29.0	11.55	36.0	9.30	43.0	7.78
22.5	14.89	29.5	11.35	36.5	9.17	43.5	7.70
23.0	14.56	30.0	11.16	37.0	9.05	44.0	7.61
23.5	14.25	30.5	10.89	37.5	8.93	44.5	7.53
24.0	13.95	31.0	10.80	38.0	8.81		
24.5	13.67	31.5	10.63	38.5	8.70		

Cane Sugar.—This is obtained by difference, deducting the milk solids (the sum of the milk sugar, proteids, fat, and ash) from the total solids first obtained.

Fat in Sweetened Condensed Milk.—Judgment as to the quality of a given brand of condensed milk is naturally based more on its fat content than on any other one factor, in that, of all its constituents, the fat is the only one that can conveniently be tampered with to the detriment of its value as a food. Hence, an accurate method for the determination of the most important ingredient, the fat, is of great importance.

The Babcock process without modification cannot be used, on account of the charring by the sulphuric acid acting on the cane sugar.

The Adams-Soxhlet method is unreliable, because the large amount of cane sugar is again a disturbing factor, enclosing the fat particles so firmly, when dried on the extraction coil, as to render its complete removal by the extracting ether difficult if not impossible.* In 1895 the writer's method described on page 149 was devised, and with certain minor modifications has been used ever since with highly satisfactory results, proving itself to be not only much quicker than the Adams-Soxhlet extraction method and easier of manipulation, but, indeed, more accurate, by reason

* When ordinary ether is used for the Soxhlet extraction, the results may not *appear* too low because the alcohol and water present in the ether dissolve not only fat but also sugar, which goes in with and is weighed as fat. With ether carefully dehydrated and freed from alcohol or with benzine or petroleum ether, the fat results will always be found far too low when the extraction is conducted under ordinary conditions.

of the fact that the cane sugar with all its attendant troubles is first eliminated.*

Calculation of Fat in Original Milk.—The "fat in the original milk," as expressed in the tables on page 147, was calculated by assuming a percentage of solids not fat of 9.3 in the original milk, this being the standard fixed by the Massachusetts law. Calculate first the fat and the milk solids to the basis of the cane-sugar-free sample. This is done by dividing the per cent of each as found in the sample by 100 less the percentage of cane sugar, and multiplying the result by 100. Ascertain the difference between the milk solids and the fat thus obtained in the cane-sugar-free sample, and divide this percentage of milk solids not fat by 9.3. The result is the "number of times condensed" (if cane sugar were not present as a diluent).

The per cent of fat in the cane-sugar-free sample, divided by the number of times condensed, as above obtained, gives the percentage of fat in the original milk.†

It is of course understood that these calculations do not give perfectly definite results. They are simply arbitrary, though they furnish quite satisfactory means of judging the quality of the milk.

If the fat in the original milk is found to be well below 3%, there is reason to suspect that skimmed milk was used in its preparation.

Other Methods for Proteids and Cane Sugar.—If desired, the proteids of condensed milk can be calculated from the total nitrogen obtained by the Gunning method as in ordinary milk (p. 109).

Bigelow and McElroy's Polarimetric Method for Cane Sugar.‡—26.048

* Parallel determinations of fat in sugar-preserved milk by the Adams-Soxhlet process, using ether carefully dehydrated and freed from alcohol, and by the writer's method involving the use of the Babcock machine, show in all cases a larger fat content by the latter or



grams of the mixed sample of condensed milk are transferred to a 100-cc. graduated sugar-flask and dissolved in water, which is boiled to make sure of normal rotation. The solution is then clarified by the addition of an acetic acid solution of mercuric iodide* and, if necessary, alumina cream, the volume is made up to 100 cc., shaken, and filtered through a dry filter. Rejecting the first part of the filtrate, a further portion is polarized. For inversion, another sample of 26.048 grams is weighed out as before and dissolved, but before clarifying, is heated to 55° C. and treated with half a cake of compressed yeast, the heating with the yeast being continued at 55° for five hours. The clarifying solution is added before cooling, and, after cooling, making up to 100 cc., and filtering as before, the invert reading is obtained with the polariscope. By this process of yeast inversion the cane sugar only is inverted, the lactose remaining unchanged. It is best to work with several samples and use the mean of the readings both for direct and invert figures. It is also best to use the double dilution method (p. 113) to compensate for the volume of the precipitated fat and proteids.

The per cent of cane sugar is calculated by the formula of Clerget,

$$S = \frac{A - B}{144 - \frac{t}{2}},$$

S being the per cent of cane sugar,
A the direct reading,
B the invert reading and t the temperature at which the observation is made.

The above process presupposes the absence of invert sugar in the sample, a supposition which Wiley claims it is fair as a rule to assume.

CREAM.

Composition.—Cream varies in composition according to the method by which it is obtained, i.e., whether (1) by allowing it to separate from the milk set in shallow pans, whence it is removed by hand-skimming, (2) by setting in deep vessels surrounded by cold water (as for example in the "Cooley" creamer) the skimmed milk being commonly drawn off from below, or (3) by the centrifugal separator. Most of the heavy cream found in the market at the present time is the product of the third or separator process.

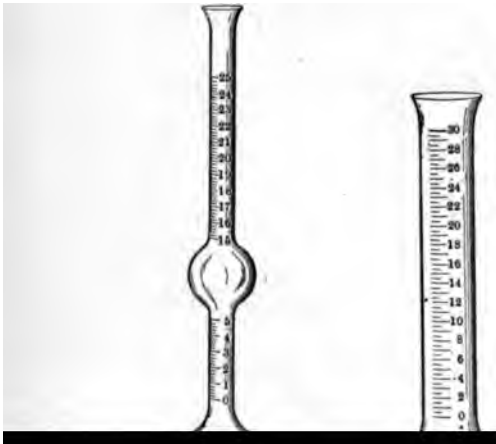
* Prepared by dissolving 53 grams of potassium iodide, 22 grams mercuric chloride, and 32 cc. of strong acetic acid in water and making up to 1 liter.

COMPOSITION OF CREAM.

Character of Cream.	Number of Analyses.	Authority.		Water.	Proteids.	Fat.	Sugar.	Ash.	Total Solids.	Solids not Fat.
By natural separation.	46	König	Mean	68.82	3.76	22.66	4.23	0.53	31.18	8.42
By centrifugal separator, "Heavy" cream.	18	Leach	Maximum	54.80	....	46.40	....	....	53.24	8.50
			Minimum	46.76	....	38.10	....	....	45.20	4.20
			Mean	51.68	....	42.02	....	....	48.32	6.30
"Light" cream.	18	Leach	Maximum	83.29	....	21.60	....	....	29.50	9.30
			Minimum	70.50	....	8.60	....	....	16.71	7.22
			Mean	77.89	....	13.86	....	....	22.11	8.25

Methods of Analysis.—The *total solids*, *ash*, *sugar*, and *proteids* are determined by similar processes to those used in milk analysis.

Fat.—The most convenient method of estimating fat is slightly modified from the regular Babcock process. The specific gravity of cream varies between such wide limits that it is best to weigh rather than measure the sample. Two varieties of cream bottle are in common use (Fig. 49) for the Babcock process, with a capacity for measuring 25 to 30 per cent of fat.



Approximately 10 grams of the well-mixed cream sample are transferred to one of these cream bottles, previously tared, and the weight of the cream accurately obtained. A convenient pipette to use for the purpose is one the end of which has been broken off to the full size

the fat is multiplied by 18 and the product, divided by the weight of cream taken, gives the per cent of fat.

U. S. Standards.*—*Standard Cream* is cream containing not less than 18% of milk fat.

Standard Evaporated Cream is cream from which a considerable portion of water has been evaporated.

Adulteration of Cream.—In some localities fat standards are fixed for cream both "heavy" and "light," those falling below such standards being deemed adulterated.

The same preservatives are employed in cream as in milk, and are

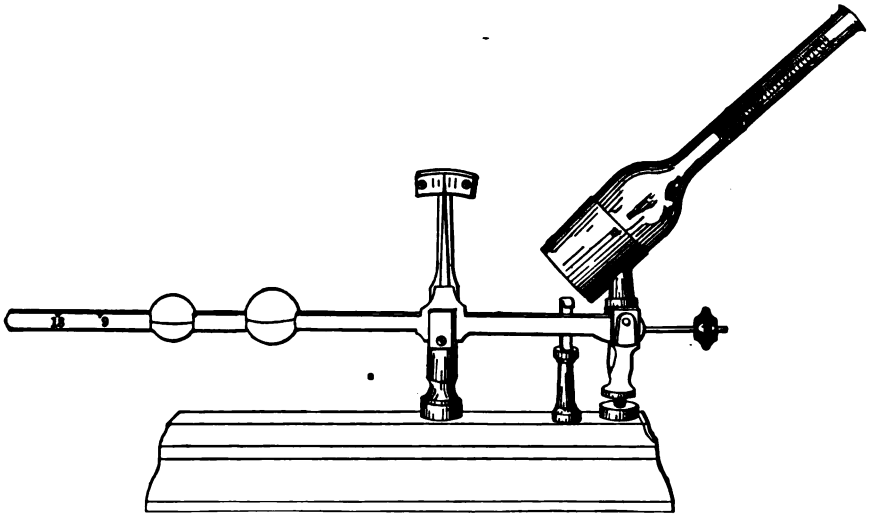


FIG. 50.—A Babcock Cream-test Scale.

detected in the same way. The color reaction for formaldehyde, by heating with hydrochloric acid and ferric chloride, is not as delicate in the case of cream as of milk, by reason of the large amount of fat. Before making the test the sample is preferably diluted with an equal volume of water, the heating is done in a casserole as with milk, but finally pour into a test-tube, and observe the color of the aqueous layer.

Gelatin in Cream.—Gelatin has been found by the writer in a number of samples of Massachusetts cream, its use being to increase the consistency. It was possible in one instance to obtain a sample of the adulterant used in the form of a powder, which proved on analysis to be chiefly gelatin

* U. S. Dept. of Agric., Off. of Sec., Circ. 10.

with a small mixture of boric acid, these ingredients serving the two-fold purpose of thickening and preserving the cream.

Gelatin is best detected in cream or milk by the method of Stokes.* He uses for reagents (1) acid nitrate of mercury, prepared by dissolving metallic mercury in twice its weight of concentrated nitric acid (sp. gr. 1.42) and diluting with twenty-five times its bulk of water, and (2) a saturated aqueous solution of picric acid.

To about 10 cc. of the cream add the same amount of the acid nitrate of mercury solution and 20 cc. of cold water. The mixture is shaken vigorously and allowed to rest for five minutes, after which it is filtered. If much gelatin is present, the filtrate will not be clear, but opalescent. To the whole or a part of the filtrate a few drops of the picric acid solution are added, and if gelatin be present in any considerable amount, a yellow precipitate is formed. Avoid an excess of acid nitrate of mercury, as this would cause a precipitate with picric acid.

If gelatin is present in small amount only, a cloudiness is produced, best seen against a dark background. In the absence of gelatin, the solution will remain perfectly clear after adding the picric acid. The reaction is delicate to 1 part of gelatin in 10,000 parts of milk or cream.

Sucrate of Lime in Cream.—In the ordinary process of pasteurizing thick cream, a process which is becoming more and more prevalent, the consistency becomes reduced, so that while the value of the cream is actually enhanced on account of its freedom from bacteria and its increased capacity for keeping, its apparent richness is impaired when compared with untreated cream of the same composition. To restore this reduced consistency, Babcock and Russell † have shown that sucrate of lime may be used to advantage. They have applied the term "viscogen" to this compound, and have suggested that cream so treated, in order to be sold



by the writer with 5 cc. of viscogen per quart was found to be 18.8, in terms of N/10 acid, added in excess and titrated back with N/10 alkali, using phenolphthalein. The alkalinity of the ash of 100 grams of pure cream (7 samples) varied between 6.4 and 13.6.

BUTTER.

The value of butter as a food depends almost entirely on its fat content, although minute quantities of protein and milk sugar are also included in its composition.

Hence butter is more logically treated in detail under the heading of fats (page 430).

CHEESE.

Nature and Composition.—Cheese consists principally of the curd and fat removed in a mass from milk, which has been curdled by the natural souring of the milk, or by the action of rennet. The separated mass of curd and fat, after being compressed, is allowed to undergo certain changes, which constitute the ripening or curing, due to the action of micro-organisms and enzymes. Sometimes cream is used as the source of cheese and sometimes skimmed milk. During the ripening process, which requires from a few weeks to several months, the characteristic flavor is developed by the changes which the proteids undergo, and the digestibility of the cheese is improved. The nature of the proteolytic changes that take place during ripening are very little understood, but a variety of complex nitrogenous products are formed, which Van Slyke divides as follows: paracasein, unsaturated paracasein lactate, paranuclein, caseoses (albumoses), peptones, amides, and ammonia. Besides nitrogenous bodies and fat, which are its chief constituents, cheese contains notable quantities of water, milk sugar, lactic acid, and mineral matter.

In some kinds of cheese salt and coloring matter are added.

Varieties.—Well-known cheeses of commerce are often named from districts, towns, or localities where they originated or are still made. They may be classified as cream, whole-milk, or skimmed-milk cheese, according to the quality of the product from which they are made, or again as hard, medium, or soft, according to whether (1) they are pressed, or (2) allowed to drain for days and sometimes weeks without pressure to a firm consistency, or (3) are made in the space of a few hours, being quickly drained on a sieve by hand pressure.

Cheddar Cheese, which is the common cheese of the United States (though originally made some 250 years ago in England and still made there), is a type of the hard cheese. *Stilton*, an English, and *Gruyère*, a Swiss cheese, belong to the medium class, and soft cheeses are represented by *Brie* and *Neufchatel*, both French cream cheeses. Other well-known varieties are *Edam*, a round, mild, long-keeping Dutch cheese, *Cammembert*, a rich cream cheese, and *Roquefort*, made originally from ewe's milk in the French town of that name, and ripened in caves in the mountains. It is flavored by a peculiar mold.

The following table, compiled by Woll,* shows the average composition of various cheeses of commerce, both foreign and domestic:

	Water.	Casein.	Fat.	Sugar.	Ash.
	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.
Cheddar	34.38	26.38	32.71	2.95	3.58
Cheshire	32.59	32.51	26.06	4.53	4.31
Stilton	30.35	28.85	35.39	1.59	3.83
Brie	50.35	17.18	25.12	1.94	5.41
Neufchatel	44.47	14.60	33.70	4.24	2.99
Roquefort	31.20	27.63	33.16	2.00	6.01
Edam	36.28	24.06	30.26	4.60	4.90
Swiss	35.80	24.44	37.40	——	2.36
Full cream, mean of 143 analyses	38.60	25.35	30.25	2.03	4.07

Van Slyke has furnished the following analysis of the nitrogen compounds in a sample of normal American Cheddar cheese six months old and cured at 60° F.:

Per Cent N in Cheese.	Per Cent Water-soluble N.	Per Cent N as Paracasein Mono-lactate.	Per Cent N as Para-nuclein.	Per Cent N as Caseoses.	Per Cent N as Peptones.	Per Cent N as Amides.	Per Cent N as Ammonia.
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]

In many localities a standard percentage for fat in cheese is fixed by law, as in the case of the U. S. standard noted above, all samples falling below that standard, unless sold as skim-milk cheese, being deemed adulterated.

Some states have specific standards for varying grades of cheese, classified as to their fat content. Thus under the Pennsylvania law* cheese is divided into five grades, as follows:

Full-cream cheese must contain not less than 32% butter fat.

Three-fourths cream cheese must contain not less than 24% butter fat.

One-half cream cheese must contain not less than 16% butter fat.

One-fourth cream cheese must contain not less than 8% butter fat.

All cheese having less than 8% fat must be branded "Skimmed Cheese."

The term "filled cheese" is commonly applied to a product in which a foreign fat, as oleo oil or lard, has been used. Filled cheese is more commonly found in localities where a carefully enforced fat-standard law prevails, but, in the absence of a standard for fat in cheese, the manufacturer can cheapen his product much more readily and conveniently by selling a skim-milk cheese in place of the whole-milk article, though not without producing a sensibly inferior product.

METHODS OF ANALYSIS.

Obtaining a Representative Sample.—*Method of the A. O. A. C.*†—By means of a cheese-trier remove, if possible, three cylindrical plugs from the cheese perpendicular to the surface and in length equal to about half the thickness of the cheese, one at the centre, one near the circumference, and one midway between the two. About one inch in length is cut off from each plug from the end having the rind, and this is discarded. The remaining portions of the plugs are then finely divided and mixed as intimately as possible.

In place of the plugs a narrow, wedge-shaped segment may be cut from the cheese, reaching from the circumference to the center, the portions near the rind being removed, and the remainder of the piece being finely divided and mixed as before. Analyses should immediately be begun after obtaining the sample.

Determination of Water.—Two or three grams of the sample are

* Penn. Laws, 1901, Act. 95, p. 128.

† U. S. Dept. of Agric., Bur. of Chem., Bul. 46, p. 55.

carefully weighed in a tared platinum dish, and dried to constant weight in an oven at 100° C. The loss of weight is reckoned as water.*

Determination of Ash.—Ignite the residue from the moisture determination at a low, red heat, cool in a desiccator, and weigh.

Determination of Fat.—*Lythgoe's Modified Babcock Method.*—Weigh accurately about 6 grams of the sample in a tared beaker. Add 10 cc. of boiling water, and stir with a rod till the cheese softens and an even emulsion is formed, preferably adding a few drops of strong ammonia to aid in the softening and emulsionizing, and keeping the beaker in hot water till the emulsion is tolerably complete and free from lumps.

If the sample is a full-cream cheese, which is usually evident from its taste and appearance, a Babcock cream-bottle is employed. The contents of the beaker, after cooling, are transferred to the test-bottle as follows: Add to the beaker about half of the 17.6 cc. of sulphuric acid regularly used for the test, stir with the rod and pour carefully into the bottle, using the remainder of the acid in two portions for washing out the beaker. Finally proceed as in the regular Babcock test for milk. Multiply the fat reading by 18 and divide by the weight of the sample taken to obtain the per cent of fat.

Werner-Schmidt Method.—Boil 2 to 3 grams of the sample in the Werner-Schmidt tube (p. 103) with 5 cc. of water and 10 cc. of concentrated hydrochloric acid till, with constant shaking, all but the fat is dissolved. Cool, add 25 cc. of ether, and shake the tube well. Draw off as much as possible of the ether, after separation, in the usual manner, and extract with four or five additional portions of the solvent.

Distil off the ether from the combined extractions, and weigh the fat.

Determination of Proteids.—From 1 to 2 grams of the cheese are treated by the Gunning method, adding after partial digestion a piece



500-cc. graduated flask. Treat the residue with repeated portions of 100 cc. each of water, heating, shaking, and decanting as above till the filtrate, or water extract, at room temperature amounts to just 500 cc. exclusive of the fat floating on top, and use aliquot parts of this water extract for the various determinations.

Water-soluble Nitrogen.—Determine the nitrogen by the Gunning method in 50 cc. of the above water extract, corresponding to 2.5 grams of cheese.

Nitrogen as Paranuclein.—Add 5 cc. of a 1% solution of hydrochloric acid to 100 cc. of the above water extract (corresponding to 5 grams of cheese), and keep the temperature at 50° to 55° till the separation is complete, as shown by a clear supernatant liquid. Filter, wash the precipitate with water, and determine the nitrogen therein by the Gunning method.

Nitrogen as Coagulable Proteids.—Neutralize the filtrate from the preceding determination with dilute potassium hydroxide, and heat at the temperature of boiling water till the coagulum,* if any, settles completely. Filter, wash the precipitate, and determine the nitrogen therein.

Nitrogen as Caseoses.—Treat the filtrate from the preceding with 1 cc. of 50% sulphuric acid saturated with C. P. zinc sulphate, and warm to about 70° C. till the caseoses settle out completely. Cool, filter, wash with a saturated solution of zinc sulphate acidified with sulphuric acid, and determine the nitrogen in the precipitate.

Nitrogen as Amides and Peptones.—Place 100 cc. of the water extract of cheese in a 250-cc. graduated flask, add 1 gram of sodium chloride and a solution containing 12% of tannin, till the addition of a drop to the clear supernatant liquid does not further precipitate. Dilute to the 250-cc. mark, shake, pour upon a dry filter, and determine the nitrogen in 50 cc. of the filtrate, which gives the amount of nitrogen in the amido-acid and ammonia compounds. Deduct from this the amount of nitrogen as ammonia separately determined, and the difference is the amido-nitrogen.

Nitrogen as peptones is obtained by subtracting the sum of the amounts of nitrogen as paranuclein, coagulable proteids, caseoses, amido-bodies, and ammonia from the total nitrogen in the water extract.

Nitrogen as Ammonia.—Distil 100 cc. of the filtrate from the above tannin-salt precipitation into standardized acid, and titrate in the usual manner.

* According to Van Slyke a precipitate at this point is rare in cheese.

Nitrogen as Paracasein Lactate.—Treat the residue insoluble in water (in obtaining the water extract, with several portions of a 5% solution of sodium chloride, to form a 500-cc. salt extract of the same, in an analogous manner to that employed in preparing the water extract. Determine the nitrogen in an aliquot part of this salt extract.

Determination of Lactic Acid.*—Add water to 10 grams of the cheese sample at 40° C. till the volume equals 105 cc. Shake and filter. Titrate 25 cc. of the filtrate (equivalent to 2.5 grams of cheese) with tenth-normal sodium hydroxide, using phenolphthalein as an indicator.

Each cubic centimeter of decinormal alkali is equivalent to 0.009 gram lactic acid.

Determination of Milk Sugar.—Boil 25 grams of finely divided cheese with two successive portions of about 100 cc. each of water, decant through a filter, and finally transfer the residue upon the filter and wash with hot water. Make up the entire aqueous extract thus obtained, when cold, to 250 cc., and determine the milk sugar by either Fehling method.

Detection of Foreign Fat.—The cheese fat, separated in the manner described below, is subjected to the various processes detailed under butter, in precisely the same way, the fat of cheese being identical with that of butter. The most ready means for judging its purity consists in determining the refraction with the butyro-refractometer, and the Reichert number.

Separation of the Fat for Examination.—Place a quantity, say 25 grams, of the finely divided sample in a large Erlenmeyer flask, add about 100 cc. of petroleum ether, cork the flask and allow it to stand for several hours with frequent shaking. Decant the petroleum ether through a filter, evaporate off the solvent by the aid of heat, and the residue will



If the fat is considerably less than the proteids, the cheese has undoubtedly been made from skimmed milk. The following analyses, made in the writer's laboratory, illustrate these grades:

Varieties of Cheese.	Water.	Fat.	Proteids.*	Ash.
Full cream (soft)	37.63	47.40	13.70	1.27
Whole milk (hard)	21.89	38.00	37.71	2.40
Whole milk (soft)	55.95	24.00	16.49	3.56
Skimmed milk (soft)	62.17	15.20	21.36	1.27
Centrifugally skimmed milk (soft)	72.80	2.00	23.52	1.68

* By difference.

REFERENCES ON MILK AND ITS PRODUCTS.*

ARKMAN. Milk, its Nature and Composition. London, 1899.

CONN, H. W. Milk Fermentations. U. S. Dept. of Agric., Off. of Exp. Station, Bul. 9.

—— Dairy Bacteriology. U. S. Dept. of Agric., Off. of Exp. Station, Bul. 25.

CONN, H. W., and ESTEN. The Ripening of Cream. Storrs Exp. Station, Annual Report, 1900.

DUCLAUX, E. Le Lait. Paris, 1894.

ELLIS and KENRICK. Milk and Milk Adulteration. Canada Int. Rev. Dept., Buls., 21, 28.

FARRINGTON, E. H., and WOLL, F. W. Testing Milk and its Products. Madison, 1898.

FLEISCHMANN, W. Lehrbuch der Milchwirthschaft. Bremen, 1893.

GERBER, N. Die Praktische Milch-Pruefung.

GROTENFELT, G. The Principles of Modern Dairy Practice. New York.

HERZ, F. J. Untersuchung der Kuhmilch. Berlin, 1889.

HUSSON, C. Le Lait. Paris, 1878.

KIRCHNER, W. Handbuch der Milchwirthschaft. Berlin, 1891.

LADD, E. F. Proteids of Cream. Jour. Am. Chem. Soc., 20, 1898, page 858.

LE CLERC, J. A. Dairy Products. U. S. Dept. of Agric., Bureau of Chemistry, Bul. 65, page 35.

LEFFMAN, H., and BEAN, W. Analysis of Milk and Milk Products. Philadelphia, 1893.

LEHMANN, J., und HEMPEL, W. Die Milchuntersuchungen. Bonn, 1894.

MACFARLANE, T. Milk and Milk Adulteration. Canada Inl. Rev. Dept., Buls. 1, 2, 9, 11, 17, 32, 43, 61, 74, 80.

—— Cheese. Canada Inl. Rev. Dept., Bul. 6.

—— Butter. Canada Inl Rev. Dept., Bul. 16.

MCGILL, A. Condensed Milk. Canada Inl. Rev. Dept., Buls. 54, 69.

OTTO, A. Die Milch und ihre Produkte. Berlin, 1892.

PEARMAIN, T. H., and MOOR, C. G. The Analysis of Food and Drugs. Part I. Milk and Milk Proteids. London, 1897.

PEARSON, R. A. National and State Dairy Laws. U. S. Dept. of Agric., Bureau of An. Ind. Bul. 26, 1900.

RICHMOND, H. D. Dairy Chemistry. London, 1889.

* For references on Butter, see p. 458.

RUSSELL, H. L. Dairy Bacteriology. Madison, 1899.
SCHOLL. Die Milch.
SCHRODT, M. Anleitung zur Prüfung der Milch u. s. w. Bremen, 1892.
SNYDER, H. The Chemistry of Dairying. Easton, 1897.
STUTZER, A. Die chem. Untersuchung der Käse. Zeits. f. anal. Chem., 1886, S. 493.
SWITHINBANK, H. Bacteriology of Milk. 1903.
TOURCHOT, A. L. Milk and Milk Adulteration. Canada Int. Rev. Bul. 53.
VAN FREUDENREICH, E. Die Bakteriologie in der Milchwirthschaft. Basel, 1893.
WANKLYN, J. A. Milk Analysis. London.
WEIGMANN, H. Die Methoden der Milchconservirung. Bremen, 1893.
WHITAKER, G. M. The Milk Supply of Boston and other New England Cities. U. S. Dept. of Agric., Bur. of An. Ind. Bul. 26, 1900.

Alabama Exp. Sta. Bul. 97. Dairy and Milk Inspection.
Annual Reports of Inspector of Milk and Vinegar, Boston, Mass.
" " " " " " Cambridge, Mass.
Arkansas Exp. Sta. Bul. 45. Milk, its Decomposition and Preservation.
Dairy Products. U. S. Dept. of Agric., Div. of Chem., Bulletin 13, part 1, 1887.
Die Milchzeitung. Bremen, 1872 et seq.
Farmers' Bulletin, No. 2. Bacteria in Milk.
" " 20. Milk Fermentation and its Relations to Dairying.
" " 29. Souring of Milk.
" " 42. Facts about Milk.
" " 74. Milk as Food.
" " 166. Cheese-making on the Farm.
Kansas Exp. Sta. Bul. 88. Keeping Milk in Summer.
Maine Exp. Sta. Bul. 23 (New Series). Cream Preservation.
Massachusetts State Board of Health Reports, 1883 et seq.
Michigan Exp. Sta. Bul. 140. Ropiness in Milk.
Minnesota Exp. Sta. Bul. 74. Milk and Cheese, Digestibility and Food Value.
New York (Geneva) Exp. Sta. Bul. 70. Reasons for Changing Milk Standards.
" " " " " " 215. Estimation of Proteolytic Compounds in Cheese and Milk.



CHAPTER VII.

FLESH FOODS.

MEAT.

General Structure and Composition.—Meat is structurally made up of muscle fibers, held together by connective tissue, through which fat cells are usually more or less abundantly distributed. Each muscle fiber has a sheath or covering known as *sarcolemma*, formed of an albuminoid substance similar to elastin, and within the fibers are contained the meat juices, which are solutions in water of proteids, non-proteid-nitrogenous extractives, and salts. The substance of the connective tissue is made up largely of the albuminoids *elastin* (insoluble) and *collagen*, the latter being convertible by boiling with water or treatment with acids into gelatin. The proteids of the meat juices consist chiefly of the globulin *myosin* (by far the most abundant), *muscle albumin*, and the muscle pigment *hæmoglobin*, or a substance closely analogous thereto.

In the living muscle there are no peptones, but the ferment pepsin is present. After death, by the action of the pepsin in presence of lactic acid, a portion of the normal proteids of the muscle undergoes, as it were, digestion, so that in meat both peptones and proteoses * are found.

The non-proteid-nitrogenous extractives are mainly *creatin*, *xanthin*, *hypoxanthin*, and *carnin*, which, from their basic character, are known as flesh bases.

The approximate proportions in which the chief constituents are present in meat is thus shown by König:

Water		75.0	to 77.0
Nitrogenized compounds	Sarcolemma (muscle fiber)	13.0	to 18.0
	Connective tissue	2.0	to 5.0
	Albumin	0.6	to 4.0
	Creatin	0.07	to 0.34
	Hypoxanthin	0.01	to 0.03
	Creatinin		
	Xanthin	Undetermined	
	Inosinic acid		
	Uric acid		
	Urea	0.01	to 0.03

* A proteose or albumose known as myoalbumose normally exists in the live muscle.

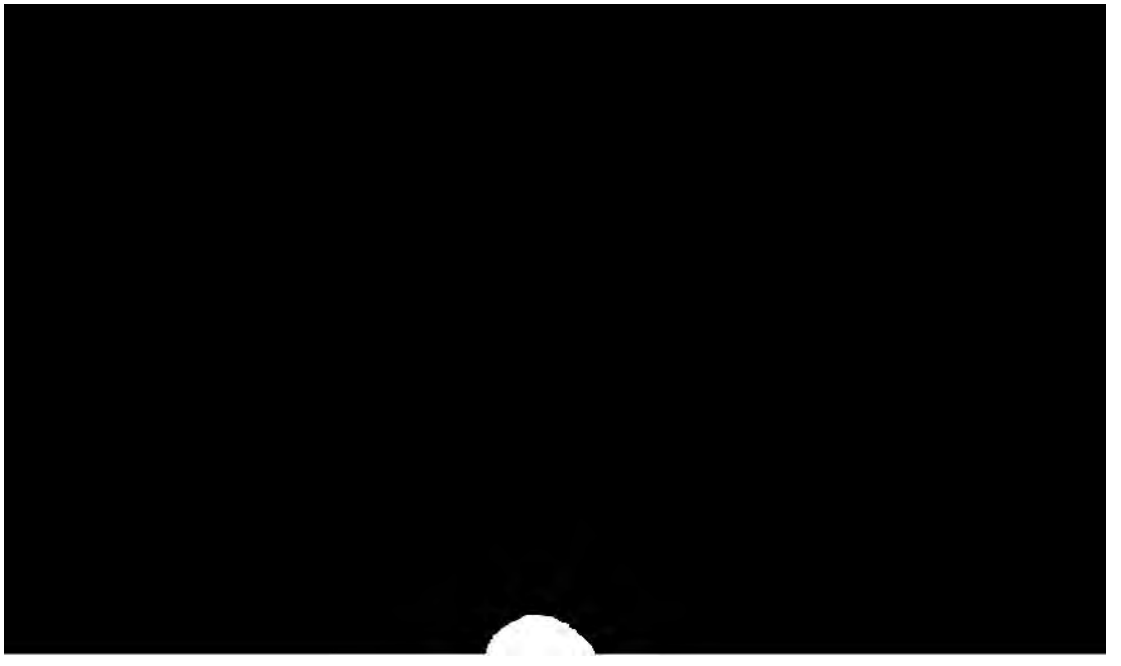
Fat		0.5 to 3.5
Other nitrogen-free compounds	Lactic acid	0.05 to 0.07
	Butyric acid, Acetic acid, Formic acid, Inosite	Undetermined
	Glycogen	(0.3 to 0.5)
Salts		(0.8 to 1.8)
Composed of:		
	Potash	0.40 to 0.50
	Soda	0.02 to 0.08
	Lime	0.01 to 0.07
	Magnesia	0.02 to 0.05
	Oxide of iron	0.003 to 0.01
	Phosphoric acid	0.40 to 0.50
	Sulphuric acid	0.003 to 0.04
	Chlorine	0.01 to 0.07

Nitrogen compounds constitute by far the most abundant and important portion of the substance of lean meat. Carbohydrates are almost entirely lacking, the small amount of glycogen and muscle sugar together constituting rarely more than 1 per cent.

Glycogen ($C_6H_{10}O_5$), sometimes called animal starch, is a white, amorphous, tasteless, and odorless substance, when pure, much resembling starch. It is soluble in water, forming an opalescent solution, and is insoluble in ether and alcohol. With iodine a port-wine color is produced, which disappears on heating and reappears on cooling. Glycogen is strongly dextro-rotary. It is converted to dextrose by boiling with dilute mineral acid.

Muscle Sugar is either entirely absent in the living muscle, or exists in traces only. After death it is formed presumably from the glycogen, and exists in a very minute quantity, probably as dextrose.

Inosite ($C_6H_{12}O_6 + H_2O$) is found in traces in the muscular substance of the heart, liver, kidneys, and testicles.



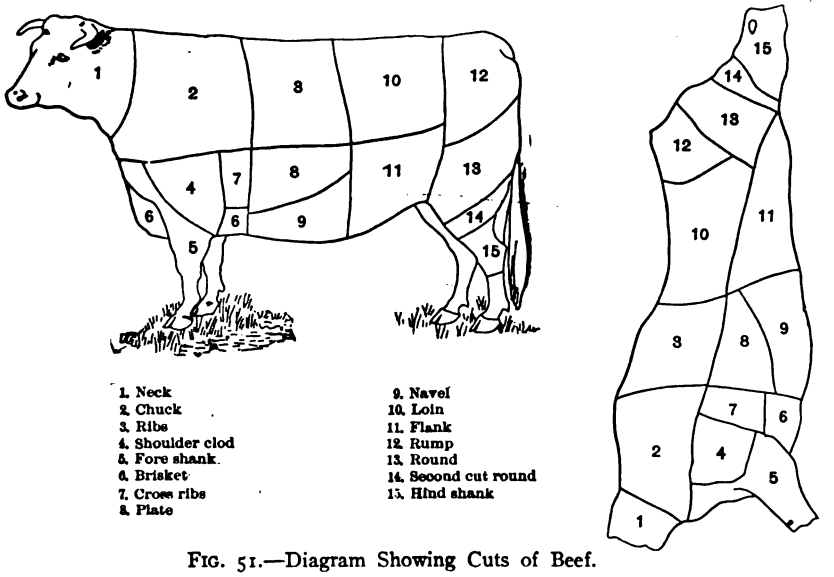


FIG. 51.—Diagram Showing Cuts of Beef.

COMPOSITION OF BEEF.

Cut.			Number of Analyses.	Refuse.	Water.	Protein.		Fat.	Ash.	Fuel Value per Pound. Cals.
						N × 6.25.	By Difference.			
Chuck:	Lean—	edible portion. .	2	.....	71.3	20.2	19.5	8.2	1.0	720
		as purchased. ..	2	19.5	57.4	16.3	15.7	6.6	0.8	580
	Medium—	edible portion. .	4	.....	68.3	19.6	18.9	11.9	0.9	865
		as purchased...	4	15.2	57.9	16.6	16.0	10.1	0.8	735
	Fat—	edible portion. .	4	.....	62.3	18.5	18.0	18.8	0.9	1135
		as purchased...	3	14.7	53.3	15.9	15.4	15.9	0.7	965
Ribs:	Lean—	edible portion. .	6	.....	66.0	16.5	16.9	9.8	0.8	790
		as purchased...	6	22.6	52.6	15.2	14.8	9.3	0.7	675
	Medium—	edible portion..	15	.....	55.5	17.5	17.0	26.6	0.9	1450
		as purchased...	15	20.8	43.8	13.9	13.5	21.2	0.7	1155
	Fat—	edible portion..	9	.....	48.5	15.0	15.2	35.6	0.7	1780
		as purchased...	8	16.8	39.6	12.7	12.4	30.6	0.6	1525
Loin:	Lean—	edible portion..	12	.....	67.0	19.7	19.3	12.7	1.0	900
		as purchased...	11	13.1	58.2	17.1	16.7	11.1	0.9	785
	Medium—	edible portion..	32	.....	60.6	18.5	18.2	20.2	1.0	1190
		as purchased...	32	13.3	52.5	16.1	15.8	17.5	0.9	1040
	Fat—	edible portion..	6	.....	54.7	17.5	16.8	27.6	0.9	1490
		as purchased...	6	10.2	49.2	15.7	15.0	24.8	0.8	1305
Rump:	Lean—	edible portion..	4	.....	65.7	20.9	19.6	13.7	1.0	965
		as purchased...	3	14.0	56.6	19.1	17.5	11.0	0.9	820
	Medium—	edible portion..	10	.....	56.7	17.4	16.9	25.5	0.9	1400
		as purchased...	10	20.7	45.0	13.8	13.4	20.2	0.7	1110
	Fat—	edible portion..	5	.....	47.1	16.8	16.4	35.7	0.8	1820
		as purchased...	5	23.0	36.2	12.9	12.6	27.6	0.6	1405
Round:	Lean—	edible portion..	31	.....	70.0	21.3	21.0	7.9	1.1	730
		as purchased...	29	8.1	64.4	19.5	19.2	7.3	1.0	670
	Medium—	edible portion..	18	.....	65.5	20.3	19.8	13.6	1.1	950
		as purchased...	14	7.2	60.7	19.0	18.3	12.8	1.0	895
	Fat—	edible portion..	5	.....	60.4	19.5	19.1	19.5	1.0	1185
		as purchased...	3	12.0	54.0	17.5	17.1	16.1	0.8	1005

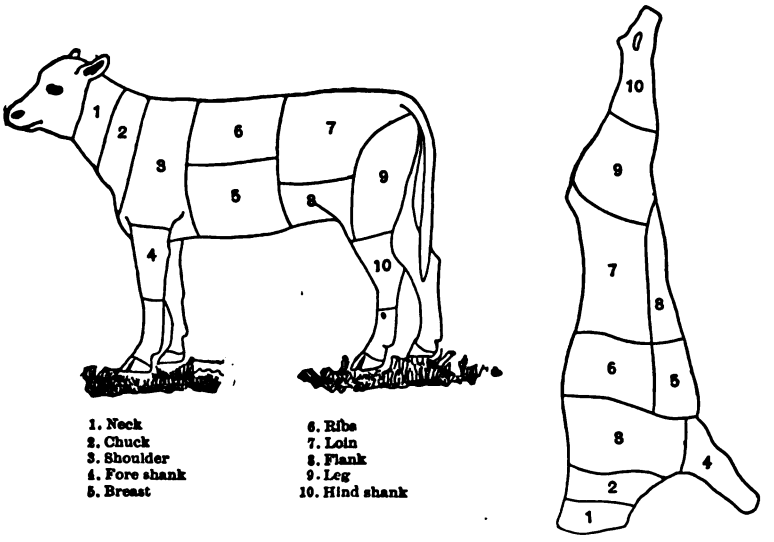


FIG. 52.—Diagram Showing Cuts of Veal.

COMPOSITION OF VEAL.

Cut.		Number of Analyses.	Refuse.	Water.	Protein.		Fat.	Ash.	Fuel Value per Pound Cals.
					N × 6.25.	By Difference.			
Chuck: Lean—	edible portion..	1	.....	76.3	.....	20.6	1.9	1.2	465
	as purchased...	1	19.0	61.8	.....	16.7	1.6	0.9	380
Medium—	edible portion..	6	.....	73.3	19.7	19.2	6.5	1.0	640
	as purchased...	6	18.0	59.5	16.0	15.6	5.2	0.8	515

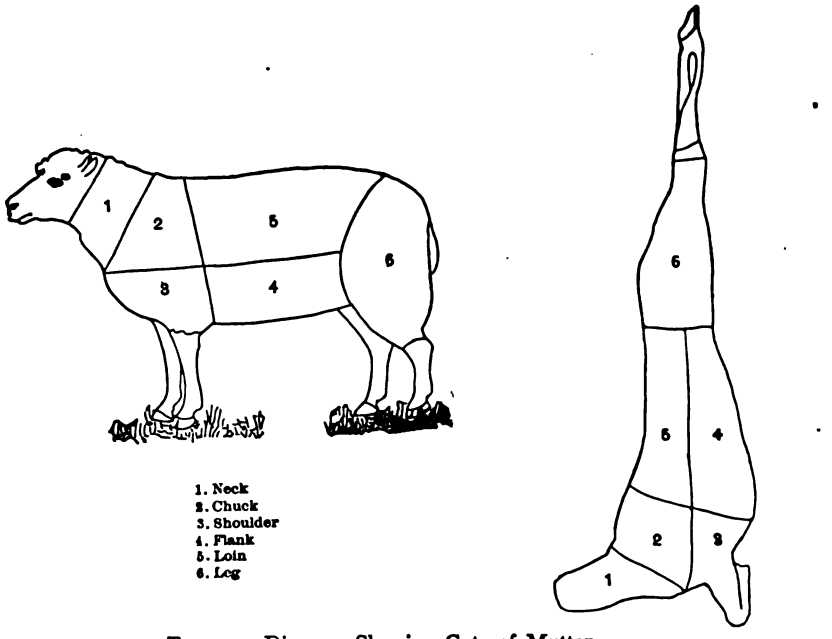
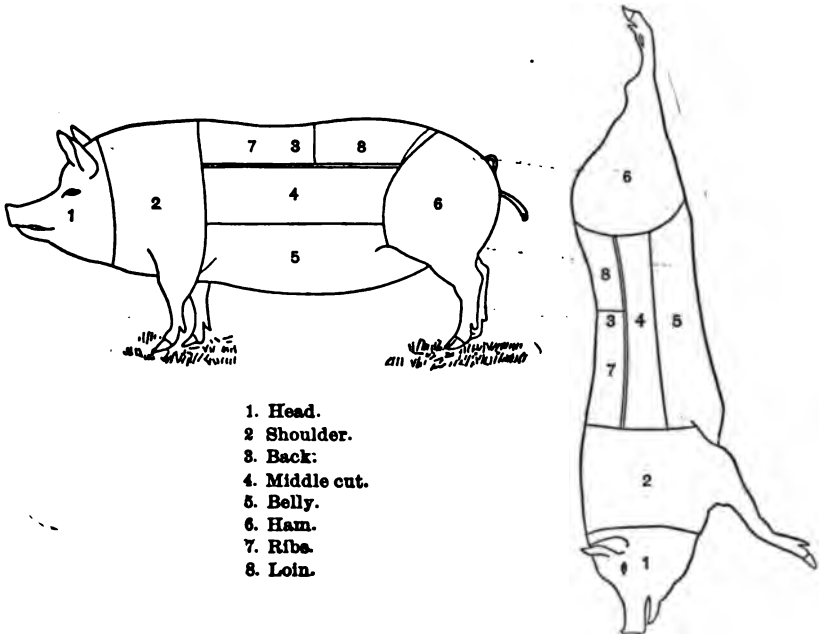


FIG. 53.—Diagram Showing Cuts of Mutton.

COMPOSITION OF MUTTON AND LAMB.

Cut.			Number of Analyses.	Refuse.	Water.	Protein.		Fat.	Ash.	Fuel Value per Pound. Cals.
						N × 6.25.	By Difference.			
	MUTTON.									
Chuck:	Lean—	edible portion..	1	.....	64.7	17.8	18.1	16.3	0.9	1020
		as purchased...	1	19.5	52.1	14.3	14.5	13.1	0.8	820
	Medium—	edible portion..	6	.....	50.9	15.1	14.6	33.6	0.9	1700
		as purchased...	6	21.3	39.9	11.9	11.5	26.7	0.6	1350
	Fat—	edible portion..	2	.....	40.6	13.9	13.7	44.9	0.8	2155
		as purchased...	2	16.5	33.8	11.6	11.5	37.5	0.7	1800
Loin:	Medium—	edible portion..	13	.....	50.2	16.0	15.9	33.1	0.8	1695
		as purchased...	12	16.0	42.0	13.5	13.0	28.3	0.7	1445
	Fat—	edible portion..	3	.....	43.3	14.7	14.2	41.7	0.8	2035
		as purchased...	3	11.7	38.3	13.0	12.5	36.8	0.7	1795
Flank:	Medium—	edible portion..	8	.....	46.2	15.2	14.8	38.3	0.7	1900
		as purchased...	2	9.9	39.0	13.8	13.6	36.9	0.6	1815
Leg:	Lean—	edible portion..	3	.....	67.4	19.8	19.1	12.4	1.1	890
		as purchased...	3	16.8	56.1	16.5	15.9	10.3	0.9	740
	Medium—	edible portion..	11	.....	62.8	18.5	18.2	18.0	1.0	1105
		as purchased...	11	18.4	51.2	15.1	14.9	14.7	0.8	900
	LAMB.									
Chuck:		edible portion..	1	.....	56.2	19.1	19.2	23.6	1.0	1350
		as purchased...	1	19.1	45.5	15.4	15.5	19.1	0.8	1090
Leg:	Medium—	edible portion..	2	.....	63.9	19.2	18.5	16.5	1.1	1055
		as purchased...	2	17.4	52.9	15.9	15.2	13.6	0.9	870
	Fat—	edible portion..	1	.....	54.6	18.3	17.1	27.4	0.9	1495
		as purchased...	1	13.4	47.3	15.8	14.8	23.7	0.8	1295
Loin:		edible portion..	4	.....	53.1	18.7	17.6	28.3	1.0	1540
		as purchased...	4	14.8	45.3	16.0	15.0	24.1	0.8	1315



1. Head.
2. Shoulder.
3. Back:
4. Middle cut.
5. Belly.
6. Ham.
7. Ribs.
8. Loin.

FIG. 54.—Diagram Showing Cuts of Pork.

COMPOSITION OF PORK, POULTRY, AND GAME.

Cut.	Number of Analyses.	Refuse.	Water.	Protein.		Fat.	Ash.	Fuel Value per Pound Cals.
				N × 6.25.	By Difference.			
PORK.								
Shoulder: edible portion..	19	.....	51.2	13.3	13.8	34.2	0.8	1690
as purchased...	19	12.4	44.9	12.0	12.2	29.8	0.7	1480

Characteristics of Sound Meat.—The reaction of meat should be acid. If neutral or alkaline, decomposition is indicated, except that alkalinity may be due to the use of alkaline salts as preservatives.

Letheby * gives the following characteristics of sound, fresh meat. In color it is neither pale pink nor deep purple, the former indicating that the animal was affected with some disease, and the latter that it died a natural death, and was not slaughtered. In appearance it is marbled, due to the presence of small veins of fat distributed among the muscles. In consistency it is firm and elastic to the touch, and should hardly moisten the finger; a wet, sodden, or flabby consistency with a jelly-like fat is indicative of bad meat. As to odor, it is practically free; whatever odor there is should not be disagreeable; a sickly or cadaverous smell is indicative of diseased meat. After standing for a day or so, it should not become wet, but on the contrary should grow drier. When dried at 100° C. it should not lose more than 70 to 74 per cent in weight; unsound meat frequently loses 80% or more. It should shrink very little in cooling.

Inspection of Meat.—While carefully drawn laws exist almost everywhere relating to the sale of meat, and government inspectors are appointed to carry out the requirements of the laws, yet in this country there is undoubtedly some meat unfit for food on the market, owing to the small number of inspectors, and the consequent comparative safety with which unscrupulous dealers may sell meats forbidden by law and escape detection. A far more rigid system of meat inspection is in force in Europe than in the United States, especially in Germany.

Unwholesomeness of Meat may be due to a diseased condition of the animal while alive, or to poisonous or injurious toxins developed by the action of bacteria after death. In the first case, the diseased conditions may be due to temporary causes only, or to the presence of animal parasites, such as trichinæ in pork, or as the result of pathogenic bacteria, causing such serious diseases as tuberculosis, anthrax, glanders, etc. It thus requires much skill and judgment on the part of the meat-inspector to correctly pass upon the suitability for food of the various meats as they appear on the market. Coplin and Bevan † give in detail useful data regarding the inspection of meat, as well as of the animal before slaughtering, showing the requisite size, weight, age, conditions of health etc., that should obtain. The detailed physical and microscopical examination involved in such inspection is, however, rarely germane to the work of the public food analyst, and will not be treated of in this manual.

* Lectures on Food, p. 210. † Practical Hygiene, pp. 130–157.

It is also beyond the scope of the present work to treat of the harmful toxins developed by bacterial action in meat and fish, causing what is known as ptomaine poisoning. The work of detecting and isolating such poisons comes within the province of the bacteriologist and biologist, rather than that of the chemist, involving many experiments upon guinea-pigs, rabbits, or other animals not usually found in the chemist's laboratory. It has furthermore been recently shown by Vaughn and Novy * that even when these toxins are present in foods in sufficient quantity to produce serious results, very considerable amounts of the food must be taken in order to isolate them by chemical means, more, in fact, than is usually available for analysis.

For the general inspection of meats for animal parasites, poisonous toxins, etc., the reader is referred to such works as those of Vaughn and Novy, Fischöder, Walley, Andrews, Cobbold, and Salmon as cited in the references on page 202.

U. S. Standards.†—*Standard Meat* is any sound, dressed, and properly prepared edible part of animals in good health at the time of slaughter. The term "animals" as herein used includes not only mammals, but fish, fowl, crustaceans, mollusks, and all other animals used as food.

Standard Fresh Meat is meat from animals recently slaughtered, or preserved only by refrigeration.

Standard Salted, Pickled, and Smoked Meats are unmixed meats preserved by salt, sugar, vinegar, spices, or smoke, singly or in combination, whether in bulk or in packages.

Standard Manufactured Meats are meats not included in the above divisions, whether simple or mixed, whole or comminuted, with or without the addition of salt, sugar, vinegar, spices, smoke, oils, or rendered fat,



Refrigeration may consist (1) in actually freezing the meat, in which condition it may be kept without decomposition almost indefinitely, until finally thawed for use, or (2) in keeping the meat at or near the temperature of freezing without actually congealing it, as is done by the use of the ordinary refrigerator. The second method, while much less efficacious than the first, serves to prevent decomposition for a considerable time; it furthermore does not impair the flavor of the meat to any such extent as the method of actual freezing.

Curing consists in subjecting the meat to various processes of drying, smoking, pickling, corning, etc., or to a combination of these processes. In simple *drying*, the meat is subjected to the heat of the sun or to artificial heat. In *smoking*, which is commonly practiced on beef and ham, the meat, which may or may not be first salted or otherwise treated, is exposed for some time to the smoke of burning beech or hickory wood, during which it becomes to some extent impregnated with the antiseptic properties of the creosote and pyroligneous acid, at the same time being dried by the heat of the burning wood. In some cases best results are obtained by a slow smoking at a comparatively low temperature, while in others quick, hot smoking is found most efficacious. The character of the meat is decidedly changed by smoking, and, according to Utescher, smoked meat is always alkaline in reaction. In *pickling*, the meat may be treated with dry salt and subjected to pressure, so that the meat juice forms the liquid for the brine, in which it is allowed to remain for some time; or, as in the ordinary process of *corning*, the beef is soaked for some days in a strong solution of salt to which a little saltpetre (KNO_3) has been added. In the process of pickling, the salts from the brine slowly diffuse into the interior of the meat by osmosis, a part of the soluble albumin passing out into the brine. The effect of the saltpetre is to preserve the natural red color of the meat, which by the action of salt alone becomes destroyed, or at least impaired.

Bacon and ham are frequently cured by pickling in brine containing salt, saltpetre, and cane sugar, and sometimes also such antiseptics as boric acid and calcium bisulphite. A common pickling fluid used for this purpose consists of 55 lbs. salt, 5 lbs. saltpetre, 5 lbs. antiseptic (boric acid), and 5 lbs. cane sugar, in sufficient water to make up 20 gallons.

The curing of bacon is sometimes effected by injecting the pickling fluid into the tissues with a "pickle-pump," capable of exerting a pressure of 40 lbs. to the square inch, and provided with a hollow, perforated needle-nozzle, which penetrates the flesh. After pickling, the bacon or ham

may be simply dried, or, if desired, smoked. Oak sawdust is frequently burned for the smoking of ham.

The Use of Antiseptics in Meat.—Most of what might be termed the modern preservatives are to be looked for in one or another of the various meat preparations, though some are better adapted than others for use in particular cases, as will be seen by reference to the composition of typical commercial preservative mixtures given on page 663.

Boric Acid and Borax, usually in mixture, are by far the most popular and widely used preservatives in connection with meat. Like salt, they are used commonly in surface application, in the case of large cuts of meat, or by mixing, in the case of sausage meat. A more recent method of application consists in impregnating the tissue of the meat with a solution of the boric mixture, by means of the above-described pickle-pump. In the curing of bacon, boric acid and borax are of late being used, to displace part of the salt, thus yielding, it is claimed, a softer, fresher, and more palatable product.

Sulphurous Acid.—As much as 1% of a solution of sulphurous acid may be added to meat without being apparent to the taste or smell. Mitchell quotes Fischer as having found that 50% of the preserved meat products (sausages, etc.) sold in Breslau in 1895 contained sulphites, varying in amount from 0.01 to 0.34 per cent of sulphur dioxide. Calcium bisulphite is a salt commonly employed.

Salicylic Acid is not of such common occurrence in meat products as the other antiseptics mentioned. The writer has found it in prepared mince-meat.

The toxic effects of the presence of these and other antiseptic chemicals in meats, and the most practical means of controlling their use are questions in controversy, presenting no new phases that have not been

ing water, there is less loss of soluble matter by reason of the formation of a more or less impenetrable coating on the outside, by the coagulation of the albuminoids. Meat that is boiled becomes softer, owing to a partial dissolving of the gelatin formed. In the dry cooking of meat, as by broiling or roasting, there is usually a hardening of the tissues, and a driving out of some of the meat juices, which are however, often recovered in the form of gravies.

Canning of Meat.—By far the most effective method of preserving meat and meat preparations of all kinds for long periods of time consists in the application of the principle of sterilizing by heat, and sealing in air-tight cans. The process of canning cooked meat and its products does not differ materially from that employed in the similar preparation of vegetables. (See Chapter XIX.) Previous to canning, the meats are usually cooked by roasting, boiling, or steaming, during which process the changes described in the preceding paragraph take place.

Many brands of canned beef, veal, tongue, ham, chicken, fowl, and game found on the market are honest preparations, and true to the names on their labels. On the other hand, there are constantly to be found examples of substitution of one meat for another, especially in spiced or highly seasoned chopped mixtures. Many varieties of the so-called potted and devilled meats and game, for example, often consist wholly or in part of a much cheaper variety than that specified on the label. The substitution of the fat of cheaper grades of meat, such as that of beef and pork in patés and purées of fowl and game, is also common. Indeed, it is largely among the canned meats and prepared meat products that instances of adulteration are to be found, since the fresh meats in whole cuts, excepting in so far as treatment with preservatives is concerned, rarely come into the hands of the food analyst for examination as to purity.

Preservatives are sometimes added to canned meats, especially in the case of dried and smoked beef, ham and bacon, and in the potted and devilled mixtures. Boric acid, benzoic acid, and sulphites have been found in these preparations.

Composition of Canned Meats.—The following table, compiled from results published by Bigelow and others,* shows the composition of various of the most common canned and preserved meats and meat products, and in one or two instances fresh meat has been included for comparison.

* U. S. Dept. of Agric., Bur. of Chem., Bulletin 13, part 10.

COMPOSITION OF CANNED MEATS.

	Number of Analyses.	Water.	Fat.	Nitrogen.				Nitrogenous Substances.				Total Ash.	Sodium Chloride.
				Total.	Insoluble in Hot Water.	Precipitated by Bromine.	Meat Bases.	Protein N × 6.25.	Proteids Insoluble in Hot Water (Coag.).	Gelatinoids & Proteids Ppt'd by Bromine.	Meat Bases.		
.....	5	68.19	12.60	3.02	2.41	0.19	0.43	18.89	15.08	1.21	1.29	0.96	0.08
um...	.....	71.17	15.33	3.19	2.52	0.24	0.53	19.94	15.75	1.50	1.65	1.13	0.24
m. ..	.....	65.81	9.89	2.83	2.32	0.14	0.33	17.69	14.50	0.87	1.02	0.78	Trace
.....	22	58.89	13.99	4.15	3.09	0.57	0.49	25.95	19.29	3.59	1.58	1.28	0.53
um...	.....	66.39	31.78	5.51	4.47	1.16	1.03	34.44	27.94	7.25	3.21	3.51	2.51
m. ..	.....	45.35	5.89	3.31	1.98	0.24	0.09	20.69	12.38	1.50	0.62	0.65	0.04
.....	11	56.35	11.43	4.26	3.58	0.32	0.36	26.63	22.37	2.02	1.11	4.69	3.37
um...	.....	60.10	22.68	4.89	4.33	0.53	0.47	30.56	27.06	3.31	1.47	7.38	4.68
m. ..	.....	46.94	6.33	3.72	3.01	0.20	0.20	23.25	18.81	1.25	0.62	3.57	2.56
.....	11	47.42	7.46	5.21	4.22	0.15	0.85	32.59	26.41	0.92	2.63	12.51	9.67
um...	.....	59.58	12.37	6.49	4.91	0.37	1.29	40.56	30.69	2.31	4.02	17.30	11.33
m. ..	.....	39.22	4.59	4.00	3.30	0.06	0.59	25.00	20.62	0.38	1.59	9.58	7.15
.....	16	69.81	9.61	3.11	2.37	0.20	0.55	19.47	14.83	1.23	1.70	1.01	0.01
um...	.....	76.91	33.66	3.60	2.97	0.36	1.22	22.50	18.56	2.25	3.81	1.27	0.09
m. ..	.....	52.16	1.24	2.13	1.33	0.12	0.15	13.31	8.31	0.75	0.47	0.63	Trace
.....	13	36.77	37.81	3.11	2.38	0.20	0.54	19.43	14.86	1.22	1.67	5.88	5.34
um...	.....	53.30	69.07	6.68	4.75	0.74	1.38	41.75	29.69	4.62	4.31	19.90	17.84
m. ..	.....	15.34	16.84	1.07	0.91	0.06	0.05	6.69	5.69	0.38	0.16	0.78	Trace
.....	17	55.17	20.23	3.11	2.50	0.21	0.39	19.43	15.64	1.33	1.23	3.71	2.90
um...	.....	71.80	38.09	3.95	3.20	0.48	0.72	24.69	20.00	3.00	2.25	6.22	4.42
m. ..	.....	39.58	6.84	2.09	1.76	0.02	0.07	13.06	11.00	0.12	0.22	0.78	0.37
.....	10	60.42	16.30	3.53	3.05	0.22	0.26	22.07	19.06	1.39	0.81	1.09	0.05
um...	.....	69.06	39.31	5.14	4.45	0.76	0.46	32.12	27.81	4.75	1.44	2.61	0.13
m. ..	.....	47.46	1.82	2.14	1.77	0.11	0.06	13.37	11.06	.....	.....	0.58	Trace
.....	15	62.44	12.72	3.62	2.86	0.26	0.44	22.61	18.39	1.66	1.39	2.31	1.16
um...	.....	70.60	30.97	5.10	4.35	0.39	0.65	31.87	27.19	2.44	2.03	3.82	2.23
m. ..	.....	44.95	2.02	1.60	1.20	0.12	0.18	10.00	7.50	0.75	0.56	1.00	0.37
.....	25	58.51	21.82	2.23	1.82	0.19	0.21	13.92	11.37	1.21	0.67	2.86	1.02
um...	.....	88.61	41.34	3.86	3.86	0.62	0.42	24.12	20.75	3.88	1.31	6.37	4.68
m. ..	.....	44.02	3.53	0.52	0.38	0.03	0.04	3.25	2.38	0.19	0.12	1.35	0.04

Sausages.—*Nature and Composition.*—Sausages are made from finely chopped meat, highly seasoned with various spices, and, as usually sold, stuffed into casings made of the cleaned and prepared intestine-skin of cattle, sheep, or hogs. The meat most commonly used is pork. Sausages are frequently home-made, especially in farm communities, the chopped and seasoned meat being stuffed in cloth bags instead of casings. Any and all kinds of meat are used in sausages, and much that is undesirable and even unwholesome is undoubtedly most readily used up in this product. There is little doubt that horse meat occasionally gets into the hands of the marketmen to be worked up in the form of sausages mixed with other meat. Sausages are sometimes artificially colored, and in some cases contain so-called "fillers" in the nature of dried bread, corn and potato starch, crackers, waste biscuit, boiled rice, etc. The alleged use of these fillers is for the purpose of absorbing moisture, but they are undoubtedly used as cheapeners.

CHEMICAL COMPOSITION OF SAUSAGES.*

Kind.	No. of Analyses.	Refuse.	Water.	Protein.		Fat.	Total Carbohydrates.	Ash.	Fuel Value, Cals.
				N×6.25	By Difference.				
Farmer: edible portion...	1	.....	23.2	29.0	27.2	42.0	.....	7.6	2310
as purchased....	1	3.9	22.2	27.9	26.2	40.4	.....	7.3	2225
Pork: as purchased....	11	.....	39.8	13.0	12.7	44.2	1.1	2.2	2125
Bologna: edible portion...	8	.....	60.0	18.7	18.4	17.6	0.3	3.7	1095
as purchased....	4	3.3	55.2	18.2	18.0	19.7	.....	3.8	1170
Frankfort: as purchased....	8	.....	57.2	19.6	19.7	18.6	1.1	3.4	1170

* U. S. Dept. of Agric., Off. of Exp. Stations, Bul. 28 (Revised Ed.).

Artificial Coloring Matter in Sausages.—Owing to the rapid color changes which freshly chopped meat, especially beef and mutton, naturally undergo, it is a common practice to employ powdered nitre or saltpetre to preserve the fresh color. As much as 4 ounces of nitre to 100 lbs. of meat is sometimes used. A larger quantity would result in a shriveled appearance. The use of artificial colors is also common, in order to permanently dye the flesh a bright red, similar to the tint which the oxyhæmoglobin naturally imparts to the beef, when fresh. A variety of colors are employed for this purpose, such as red ochre, coal-tar dyes, cochineal, etc.

ANALYTICAL METHODS.

In analyzing meats and meat products due regard must be paid to their perishable nature, and, for this reason, immediately after their receipt by the analyst the various determinations should, if possible, be promptly begun and rapidly carried out. If delays are absolutely necessary, the samples, as well as some of the solutions, especially during the earlier course of the analysis, should be kept on ice to prevent decomposition. Refuse material, such as bones, skin, gristle, etc., are separated as completely as possible by means of a knife from the edible portion, and the latter, cut first into small pieces, is passed through a sausage-machine or ordinary household meat-chopper, in order to reduce to a homogeneous, finely divided mass.

Determination of Water.—From 1 to 3 grams of the finely divided material are weighed in a tared platinum dish, and dried to constant weight at a temperature of 100° C. in an air-oven. A slight oxidation of the fat may introduce a trifling error, but, excepting for the most exact work, where the drying should be accomplished in an atmosphere of hydrogen, or *in vacuo*, the above method is sufficiently close.

Determination of Ash.—The residue from the total solids is incinerated in the original dish over the free flame at a low red heat, or in a muffle, adding if necessary, at intervals, a very little powdered ammonium nitrate. A perfectly white ash is difficult to obtain.

Determination of Fat.—About two grams of the sample are carefully weighed in a tared Schleicher-and-Schull Soxhlet shell, which, after drying in the air-oven at 100°, is transferred to an extraction apparatus and subjected to continuous extraction for at least sixteen hours with anhydrous ether, or pure petroleum ether. It is impossible to extract all the fat in



FATS OF VARIOUS MEATS AND MEAT PREPARATIONS.*

		Specific Gravity $\frac{100°}{100°}$	Specific Gravity $\frac{100°}{15°}$	Degrees Butyro-refractometer at 35°.	Index of Refraction at 35°.	Melting-point.	Chilling-point.	Iodine Number.	Koettstorfer Number.	Soluble Acids.	Insoluble Acids.	Heat with H_2SO_4.
Beef, roast and boiled:	Average.....	0.8953	0.8589	52.8	1.4610	40.1	32.2	45.6	194.8	0.45	93.54	35.8
	Maximum...	0.9046	0.8679	55.5	1.4629	43.9	37.0	50.6	200.0	0.74	95.27	36.0
	Minimum ...	0.8925	0.8563	47.0	1.4571	36.5	27.8	36.1	188.0	0.22	91.90	35.6
Beef, canned, corned:	Average.....	0.8934	0.8572	53.98	1.4618	40.1	31.0	42.7	196.3	0.60	92.37	36.2
	Maximum...	0.8944	0.8581	56.0	1.4631	43.4	34.5	48.6	210.0	1.62	94.50	37.0
	Minimum ...	0.8925	0.8563	52.7	1.4609	37.2	29.9	37.9	191.0	0.09	89.51	35.5
Beef, canned, dried, and smoked:	Average.....	.....	.....	55.2	1.4627	39.3	26.0	54.1				
	Maximum...	.....	.....	58.5	1.4649	41.6	29.0	57.5				
	Minimum ...	.....	.....	51.0	1.4599	37.7	22.0	50.9				
Horse meat:	Average.....	0.9067	0.8699	62.3	1.4673	29.7	17.2	66.4	201	1.32	91.37	49.9
	Maximum...	0.9184	0.8811	76.5	1.4762	32.5	25.0	77.0	205	2.76	95.44	56.5
	Minimum ...	0.8868	0.8508	55.2	1.4625	27.2	12.0	61.4	196	0.19	88.18	46.2
Ham and bacon, canned:	Average.....	0.8893	0.8533	53.4	1.4615	27.7	20.3	57.8	193	0.98	90.80	41.4
	Maximum...	0.9066	0.8698	58.2	1.4648	30.5	24.0	68.2	207	2.63	94.51	43.5
	Minimum ...	0.8609	0.8260	49.0	1.4586	23.6	17.5	48.5	179	0.19	86.40	39.8
Tongue, canned:	Average.....	0.8929	0.8565	54.0	1.4628	35.8	26.7	48.8	193	0.97	90.91	37.8
	Maximum...	0.9096	0.8726	58.0	1.4677	41.4	33.0	63.6	205	2.60	94.78	46.5
	Minimum ...	0.8717	0.8363	47.2	1.4573	26.5	15.0	37.8	181	0.42	86.20	32.4
Fowl:	Average.....	0.8999	0.8633	56.7	1.4637	31.2	19.6	76.1	198	0.51	95.58	45.4
	Maximum...	0.9157	0.8785	62.5	1.4674	34.0	36.5	86.4	199	0.92	96.03	52.0
	Minimum ...	0.8849	0.8490	49.0	1.4590	28.0	12.0	67.0	196	0.15	95.39	38.9
Sausage, canned:	Average.....	0.8953	0.8596	54.6	1.4620	35.2	22.3	55.0	193	0.70	90.76	40.0
	Maximum...	0.9225	0.8850	60.3	1.4660	41.5	33.5	63.8	200	1.70	92.68	40.0
	Minimum ...	0.8786	0.8482	49.2	1.4559	27.6	12.0	39.8	184	0.10	88.25	40.0

* U. S. Dept. of Agric., Bur. of Chem., Bul. 13, part 10, p. 1490 et seq.

Determination of Total Nitrogen.—Two grams of the sample are treated according to the Gunning method (page 61). If by a qualitative test nitrates are present (as in the case of corned beef and similar preparations), the modified Gunning method, including the nitrogen of nitrates, is employed (page 63). While in the case of meat the time-honored custom of representing the total protein or nitrogenous substances by N×6.25 is by no means strictly accurate, considering the wide variation in nitrogenous content of the various compounds present, a fairly close approximation to the total nitrogenous substance present is obtained by using this factor, since the proteids form by far the largest group of all. The factors to be employed in the calculation of the flesh bases are given elsewhere (page 184).

Separation and Examination of Nitrogenous Bodies.—Just how far the analyst should subdivide the various nitrogenous bodies present in meat depends largely on the importance of the case in hand. Frequently the simple determination of total nitrogen as above is sufficient. It is rarely necessary to go further than to divide the nitrogenous bodies into several main groups, according to their solubility in water or other solvents, and their behavior toward certain reagents.

Knorr's Method * consists in extracting a weighed portion of the finely divided, fat-free sample with strong alcohol, which dissolves the meat bases, but not the proteids. With the meat bases are also dissolved of course the minute quantity of lactic acid, inosite, and glycogen which the meat contains. The alcoholic extract is dried and weighed, and the nitrogen determined in the residue from this extraction. A fresh portion of the sample is then extracted repeatedly with cold water, which removes both the soluble albumin and the meat bases. The residue from the water extract is then dried at 100° and weighed. On filtering the



The nitrogen may be determined separately in each of these classes, and by the approximate factor the corresponding nitrogen substance, or class of substances ascertained.

General Scheme for Separation.—In order to separate most completely the various classes of nitrogenous bodies found in meat, a portion of the fat-free sample should first be exhausted with cold water, which removes the soluble proteids (albumin, proteose, and peptones), leaving behind the insoluble globulins, the sarcolemma, the albuminoids of the connective tissue (clastin, etc., also insoluble), and the collagen. By next exhausting with boiling water the collagen is removed in the form of soluble gelatin.

By treatment of the combined aqueous extract (1) with bromine in connection with hydrochloric acid or zinc sulphate, or (2) with phosphotungstic and tannic acids, as hereafter explained, the soluble proteids, including the peptones and gelatin, may be separated from the meat bases.

In obtaining the results from which the table on page 176 was compiled,* but three divisions of nitrogenous substances were made, viz., (1) those insoluble in hot water; (2) those precipitated from the water extract by bromine; and (3) the flesh bases.

Nitrogenous Substance of the Meat Fiber. — About 2 grams of the finely divided sample accurately weighed, are first extracted with ether, and, after boiling in water for some minutes, the mixture is transferred to a Gooch crucible or filter-plate, and further extracted with hot water till all soluble matters are removed. Determine the nitrogen in the insoluble residue by the Gunning method. $N \times 6.25 =$ the nitrogenous substance of the meat fiber, including the coagulated proteids and globulins.

If the sample is first thoroughly exhausted with cold water before the hot-water extraction as above, the coagulable albumin is removed, and, if it is desired, the nitrogen may be separately determined in the cold- and hot-water extracts, thus separating the albumin from the gelatinoids.†

Determination of Proteoses, Peptones, and Gelatin.‡—The entire filtrate from the meat fiber containing all the water-soluble extractives, as obtained in the preceding paragraph, is evaporated to small volume.

* U. S. Dept. of Agric., Bur. of Chem., Bul. 65; also Bul. 13, p. 1396.

† The difference between direct extraction with hot water and successive extraction first with cold water and then with hot should be clearly borne in mind. If treated at once with hot water, the albumin is coagulated and left behind in the meat with the meat fiber and globulins, while the collagen is rendered soluble and removed. By cold-water extraction at the beginning, the albumin is removed and the collagen left behind. Treatment first with cold and then with hot water is the only means of removing *all* water-soluble material.

‡ U. S. Dept. of Agric., Division of Chemistry, Bulletin 54.

If cold water is first used for extraction before treatment with the hot water, a turbidity will result on heating, due to coagulation of the albumin. In this case the extract should be filtered, the clear solution transferred to a Kjeldahl flask and acidulated strongly with hydrochloric acid, after which 2 cc. of liquid bromine are added and the contents thoroughly shaken. If, after shaking, all the bromine has been absorbed, more should be added, so that after saturation two or three drops of bromine are left undissolved. Allow the mixture to stand over night, during which all the precipitate will have settled out. Decant the supernatant liquid through a filter, and wash the residue in the flask by shaking several times with added water, depending upon the excess of bromine to saturate the wash-water, and decanting the wash-water through the filter. Finally return the filter to the original flask with the washed residue, and determine the nitrogen by the Gunning method. $N \times 6.25$ = combined proteose, peptone, and gelatin. This method does not give absolutely accurate results, owing to the fact that bromine does not completely precipitate all the proteose and peptone. The method is convenient on account of its simplicity, and is undoubtedly approximately correct.

*Allen and Searle's Method.**—After evaporation of the water extract, and filtration, if necessary as described in the preceding paragraph, add two or three drops of dilute sulphuric acid and saturate the solution with powdered zinc sulphate,† avoiding an excess. Each 50 cc. of solution require about 80 grams of the salt. Allow the coagulated proteids to settle out, filter, and wash with a saturated solution of zinc sulphate. The filtrate from the treatment with zinc sulphate is then acidulated with hydrochloric acid in a Kjeldahl flask, treated with bromine, and the process described in the preceding paragraph carried out, adding the zinc sulphate precipitate to the residue precipitated by bromine in the



water at 98.2° C.; creatin, 1 part in 107 parts at 98.1° C.; creatinin, 1 part to 222 at 97.9°; hypoxanthin, 1 to 98 at 97.6°, etc., though all of these are precipitated by phosphotungstic acid.

Mallet's method is based on the principle that amido-bodies, by precipitating with phosphotungstic acid and washing the precipitate with hot water, can be separated from all proteid-like bodies excepting peptones. These last are, however, precipitated by tannic acid, hence the separation can be made complete.

The combined cold- and hot-water extract from the meat fiber is heated to 90° C., acidified with acetic acid, and filtered free from coagulum (albumin), which is washed with hot water. While still hot, a slight excess of tannic acid is added to the filtrate, which precipitates the peptones and gelatin.* Cool, filter, and wash with cold water.

The filtrate from the peptones is heated again nearly to boiling, and a solution of phosphotungstic acid, Reagent No. 20, is added as long as a precipitate continues to form, avoiding, however, a large excess. The precipitate is washed by hot water upon a filter, and the washing continued, taking care that the temperature of the water does not fall below 90° C.

If it is desired to separate the albumin from the other proteids, and the peptones and gelatin from the proteoses, the nitrogen may be separately determined in the three precipitates; otherwise the three are combined, and the nitrogen determined in all together. In either case the filters with their contents should be introduced into strong sulphuric acid as soon as possible after washing to avoid loss by decomposition. The factor 6.25 is employed.

Determination of Gelatin.—*Modified Stutzer's Method.*†—A weighed portion of the sample, say 10 grams, is thoroughly extracted by boiling water, the extract transferred to a porcelain dish containing about 20 grams of previously ignited sand, and evaporated to dryness. The residue is then stirred with four successive portions of absolute alcohol, using about 50 cc. each time and pouring it off through a filter consisting of a layer of asbestos fiber on a perforated porcelain plate within a funnel. This funnel is surrounded by chopped ice, and is so arranged that gentle suction may be used to hasten the filtration. The residue is then repeatedly stirred with successive portions of about 100 cc. each of a mixture containing 100 cc. of 95% alcohol, 300 grams of ice, and 600

* Peptones are not completely precipitated by phosphotungstic acid, but according to Halliburton their precipitation by tannic acid is complete.

† U. S. Dept. of Agric., Bul. 13, part 10, p. 1397.

grams of cold water, the portions being passed through the asbestos filter, and the washing being continued till the solution is colorless as it comes from the filter, keeping the temperature always below 5°. The asbestos is then transferred to a beaker with the washed residue, and the whole thoroughly extracted with boiling water. The hot-water extract is evaporated to small volume, and washed into a Kjeldahl flask, in which it is then evaporated to dryness, and the nitrogen determined by the Gunning method: $N \times 5.55 =$ gelatin.

Determination of Flesh Bases.—From the total nitrogen, determined in a portion of the original sample, is deducted the sum of the nitrogen found in the extracted meat fiber and that of the combined albumin, proteose, peptone, and gelatin. The difference is the nitrogen of the flesh bases, which, multiplied by 3.12, expresses the percentage of flesh bases calculated as creatin.

FACTORS CORRESPONDING TO FLESH BASES.*

Substance.	Formula.	Proportion of Nitrogen.	Factor.
Creatin	$C_4H_9N_3I_2$	42 in 131	3.12
Creatinin	$C_4H_7N_3I$	42 in 113	2.69
Xanthin	$C_5H_4N_4O_2$	56 in 152	2.71
Xanthocreatinin	$C_5H_{10}N_4O$	56 in 142	2.54
Hypoxanthin	$C_5H_4N_4O$	56 in 137	2.44
Carnin	$C_7H_8N_4O_3$	56 in 196	3.50
Leucin	$C_6H_{13}NO_2$	14 in 131	9.36
Tyrosin	$C_9H_{11}NO_3$	14 in 181	12.93
Urea	CH_4N_2O	28 in 60	2.14
Uric acid	$C_5H_4N_4O_3$	56 in 168	3.00

* Allen's Com. Org. Analysis, Vol. IV, p. 308.



Detection of Boric Acid.—A portion of the finely divided meat, mechanically freed from fat as far as possible, is warmed with water acidified with hydrochloric acid, and turmeric-paper is soaked in the extract. The rose-red color of the turmeric-paper after drying (turned olive by weak alkali) is indicative of boric acid.

A more delicate method of procedure consists in burning to an ash a portion of the meat, after treatment with lime water, and testing with the turmeric-paper a solution of the ash slightly acidified with hydrochloric acid.

Detection of Salicylic Acid.—The sample, mechanically freed from fat, is slightly acidified and shaken out with ether. The ether extract evaporated to dryness is tested with a drop of a solution of ferric chloride. A deep-violet coloration indicates salicylic acid.

Starch in Sausages.—*Detection.*—The addition of cracker or bread crumbs is best indicated by the presence of considerable starch, which is readily recognized by the iodine test, applied by boiling up a portion of the sample with water, cooling and adding a drop of iodine reagent to the liquid. The characteristic blue color is produced if starch be present in notable quantity. One per cent or more of starch present may be due to the pepper and spices used in seasoning the sausage. A small admixture of starch is rendered apparent when a thin section of the sausage is treated with a drop of iodine reagent and viewed under the microscope. A microscopical examination will also reveal the character of the starch, whether it is from cereals or from pepper.

Estimation of Starch.—The regular acid conversion process, p. 223, may be applied, but more accurate results are obtained by the method of inversion with malt extract. Medicus and Schwab * prepare the malt extract for this purpose by digesting 5 grams of ground malt with 50 cc. of water for one and one-half hours at 20° to 30° C. In making the starch estimation, they digest for two hours at a temperature of from 40° to 50° C. 20 grams of the sausage mixed with 20 cc. of the malt extract, and afterwards for eighteen hours at room temperature. After filtering and washing, the filtrate is boiled to coagulate the albumin and again filtered. The second filtrate is then made up to 200 cc., 20 cc. of 25% hydrochloric acid (specific gravity 1.125) are added, and the starch determined in the regular manner.

Mayrhofer's Method.†—This is considered the simplest and most

* Berichte d. chem. Gesell., XII, p. 1285.

† Zeits. Nahr. Untersuch., 1896, p. 331; Abs. Analyst, 1897, p. 11.

reliable method of estimating the starch in such substances as sausages. From 60 to 80 grams of the sample are heated on the water-bath with an 8% solution of alcoholic potassium hydroxide, which, in the case of pure sausages, dissolves nearly everything except a little cellulose. To prevent gelatinization, warm alcohol is added to dilute the solution, which is then filtered through paper or asbestos. The starch is contained in the insoluble residue, which is washed with alcohol till the washings are no longer alkaline, after which it is treated with an aqueous solution of potassium hydroxide, and the starch solution made up to a definite volume. To an aliquot part of the solution 95% alcohol is added, whereupon the starch comes down as a flocculent precipitate. This is collected on a weighed filter, washed with alcohol and ether, dried, and weighed. The filter with its contents is then burnt to an ash, the amount of which is deducted.

In order to avoid the ash determination, the starch may be precipitated from a weak acetic acid solution instead of from an alkaline solution, the potassium acetate formed being soluble in the alcohol, and nothing but pure starch is precipitated.

Characteristics of Horse Flesh.—Although certain authorities have found distinguishing characteristics in color, consistency, odor, etc., between horse flesh on the one hand, and beef and pork on the other, it is extremely difficult, by its physical properties, to detect horse flesh when mixed with other meat, especially when the mixture is chopped. Horse flesh has a much coarser texture and is darker in color than beef. The muscle fibers are, as a rule, shorter in horse flesh. On treating horse flesh with formaldehyde, Ehrlich * has found that a very characteristic odor is developed within forty-eight hours, suggestive of roasted goose flesh.

Certain of the constants of the fat of horse meat differ from those of

flesh than in other meat, renders it possible in some cases to detect horse flesh, when present in the mixture.

The following table prepared by Bujard shows the relative amount of glycogen in various kinds of meat and sausages:

	Water.	Glycogen Direct.		Glycogen in Dried Substance.	
		Niebel Method.	Mayrhofer Method.	Niebel.	Mayrhofer.
Horse flesh	74.44	0.440	0.445	1.721	1.741
"	74.87	0.600	0.520	2.388	2.069
"	76.17	1.827	1.727	7.667	7.247
"	76.00	0.592	0.610	2.466	2.542
Red sausage (Knackwurst)	69.26	.....	0.038	.....	0.124
Pork sausage	67.25	.....	0.24	.....	0.733
Veal	74.6	.....	0.086	.....	0.342
Pork	75.0	.....	0.186	.....	0.744

In beef Bujard found 0.74 and 0.073 per cent of glycogen calculated in terms of dried substance, and, in sausages made exclusively from horse meat, amounts of glycogen ranging from 0.05 to 5.34, the sample in the latter case being made from the liver. It was formerly thought possible to detect as small an amount as 5% of horse flesh in mixture, but later investigation showed that after the death of the animal, glycogen, though present at first in considerable quantity, decomposes more or less rapidly, going over into muscle sugar (dextrose). Hence, while the presence of much glycogen is suspicious, its absence is by no means proof that horse flesh was not used.

Niebel did not consider the failure of the glycogen test as sufficiently conclusive to establish the absence of horse flesh, on account of the tendency toward decomposition of the glycogen. In the absence of starch, he regards the presence of more than 1% of dextrose in the fat-free meat, after conversion of the carbohydrates, to be proof of the presence of horse-flesh.

Detection of Glycogen.—From the well-known color reaction produced by iodine on glycogen, horse flesh can often be detected, when present in sausages, unless obscured by the presence of starch or dextrin.

Brautigam and Edelmann* proceed as follows: 50 grams of the finely divided meat are boiled with 200 cc. of water for an hour, and, after cooling, dilute nitric acid is added to the broth to precipitate the proteids and to decolorize. The broth is then filtered, and a portion of the filtrate

* Pharm. Central., 1893, p. 557.

is treated in a test-tube with a freshly prepared, saturated, aqueous solution of iodine, or, better, with a mixture of 2 parts iodine to 4 parts potassium iodide and 100 parts water, the reagent being carefully added so as not to mix with the broth, but form a layer above it. If glycogen be present in considerable amount, a wine-colored ring is observable at the junction of the two layers. On heating the test-tube, the coloration disappears if due to glycogen, but it reappears on cooling. This reaction was found to occur with horse flesh and not with beef, mutton, veal, or pork.*

If the color is not clearly apparent, the chopped meat is heated on the water-bath with a solution of potassium hydroxide (using an amount of potassium hydroxide equivalent to 3% of the weight of the flesh) till the fiber is decomposed, after which the broth is concentrated to half its volume, treated with nitric acid to precipitate the proteids, filtered, and treated with the iodine solution as previously.

Determination of Glycogen.†—*Niebel's Modification of Brücke's Method.*—This method is applicable only in the absence of dextrose and dextrin. If therefore the presence and character of the starch indicates the presence to a considerable extent of cracker crumbs or other cereal "filler," the method is not accurate.

A weighed portion of the flesh is heated on the water-bath with 3 to 4 per cent of potassium hydroxide and four volumes of water for six hours. Evaporate the broth to half its original bulk, and add, after cooling, a solution of mercuric iodide in potassium iodide,‡ which precipitates the protein. Filter, and to the clear filtrate add 2½ times its volume of 95% alcohol, collect the precipitated glycogen on a filter, wash first with 60% alcohol, then with 95% alcohol, then with absolute alcohol, then with ether, and finally with absolute alcohol. Dry at 115° C. and weigh.



solve in strong acetic acid. Add to the solution, after cooling, sufficient hydrochloric acid to produce a yellow color, then pour into 2½ volumes of alcohol, and proceed as in the preceding paragraph.

Mayrhofer's Method,* on which the results in the table on page 187 are based, is as follows: Dissolve a weighed portion of the flesh in an aqueous solution of potassium hydroxide, precipitate the proteids by hydrochloric acid and Nessler's reagent, filter, and treat the clear filtrate with alcohol, which precipitates the glycogen. This is collected on a tared filter and washed, first with dilute alcohol, and finally with ether, dried at 110° C., and weighed.

Pflüger and Nerking's Method.†—Of the finely divided sample 50 grams are heated on the water-bath with 200 cc. of 2% potassium hydroxide till the solution is practically complete. After cooling, the solution is made up to 200 cc. with water, shaken, and filtered. To 100 cc. of the filtrate, 10 grams of potassium iodide and 1 gram potassium hydroxide are added, and the solution stirrred till clear, after which 50 cc. of 95% alcohol are added and the mixture allowed to stand over night. This precipitates the glycogen. Filter, and wash the precipitate with a solution made up of 1 cc. 70% potassium hydroxide, 10 grams potassium iodide, 100 cc. water, and 50 cc. 95% alcohol. After further washing the glycogen with 2 parts strong alcohol and 1 part water, dissolve in water and by means of Brücke's mercuric-iodide-in-potassium-iodide reagent (see footnote, p. 188) remove any remaining nitrogenous substances. Filter if turbid, and to the solution add common salt (about 2 milligrams per 100 cc. of solution), and reprecipitate the glycogen by adding 2 volumes of 95% alcohol. Filter, wash first with 95% alcohol containing a little common salt, then with absolute alcohol, and lastly with ether. Dry and weigh.

Bigelow suggests that the glycogen as above obtained be converted by acid hydrolysis to dextrose, which is determined in the regular manner. Dextrose × .9 = glycogen.

Identification of Raw Horse Flesh by the Blood Serum Test.‡—This test depends upon the recent development of the principle that when a rabbit has been inoculated with the blood of a particular animal, as for instance that of the horse, the serum of the rabbit's blood will react with

* Forsch. Ber., 1897, IV, 47.

† Arch. ges. Physiol., 1899, 76, 531–542; Bul. 65, Bur. of Chem., p. 13. Recommended for Provis. Adoption by the A. O. A. C.

‡ *Schütze, A.*, Ueber weitere Anwendungen der Präcipitine. (Deuts. med. Wochs., 1902, No. 45, p. 804.)

Wassermann, A., u. Schütze, A., Ueber die Entwickelung der biologischen Methode

the blood of the horse and with that of no other animal. To prepare the blood serum for a reagent, inject a rabbit with 10 cc. of defibrinated horse's blood every day for five to six days, either subcutaneously or intravenously. The blood afterwards taken from the rabbit is clotted, and the filtered serum is used in making the test, or, if the reagent is to be kept for some time, the rabbit's blood serum is dried and an aqueous solution used for the reagent.

If the clear expressed juice from the suspected flesh, filtered if necessary, be treated with a few drops of the rabbit's blood reagent, prepared as above, a cloudy precipitate will be produced in the case of horse flesh.

By inoculating different rabbits in like manner with the blood of various animals, the flesh of the corresponding animals may be recognized from the reaction of the blood serum of the rabbit with its juices. Only raw flesh responds to the test, as heating destroys the virtue of the reagent.

Determination of Muscle Sugar (*Dextrose*).—Boil a weighed quantity of the finely divided sample, say 50 grams, with water, add an excess of normal lead acetate solution, and make up with water to a given volume, say 250 cc. Filter, and to an aliquot part of the filtrate add enough of a saturated solution of sodium sulphate to precipitate the lead. Again filter, make up to a given volume, and determine the dextrose in a measured part of the solution by either of the regular methods.

Detection of Coloring Matter.—*Red Ocher* is indicated by an excessive amount of iron in the ash.

Cochineal is most readily tested for by the method of Klinger and Bujard.* The sausage, finely divided, is heated with two volumes of a mixture of equal parts of glycerin and water for several hours on the water-bath, the mixture being slightly acidified. The yellow solution is passed through a wet filter, and the coloring matter, if present, is pre-

through the spectroscope for the characteristic absorption-bands of carmine lake, lying between *b* and *D*.

Spaeth * has shown that both *carmine* (cochineal) and *anilin red*, which are the dyes most commonly used for coloring sausages, can be most readily extracted therefrom by warming the finely divided material a short time on the water-bath with a 5% solution of sodium salicylate.

Vegetable and Coal-tar Colors.—Various solvents are suggested for the removal of these dyes from sausage meat. Allen † recommends extraction with methylated spirit (a mixture of ethyl alcohol with 10% methyl); Bigelow ‡ recommends heating with a mixture of 50% glycerin slightly acidified; A. S. Mitchell uses alcohol acidified with hydrochloric acid; Spaeth a 5% solution of salicylate of soda. Other solvents applicable in some cases are dilute ammonia and amyl alcohol. The solvent, after filtering, is evaporated to small volume, acidified with hydrochloric acid, and white wool is boiled in it. If the wool is distinctly dyed, a coal-tar color is undoubtedly present, and this can often be identified by methods given in Chapter XVI. According to Marpmann, pure normal flesh containing natural color only is completely decolorized by macerating for two hours in 50% alcohol, while artificially colored meat remains colored after this treatment.

Marpmann's Microscopical Method.§—Moisten a thin section of the sausage with 50% alcohol, and examine under the microscope. Some colors are readily apparent without further treatment. If only traces of color are present, clarify the substance by treatment with xylol, which is removed by the use of carbon tetrachloride. The mass rendered transparent by this treatment is then immersed in cedar oil and examined, the coloring matters, if present, being especially apparent. If the color used is fuchsin (magenta), carmine, logwood, or orchil, the substance of the cell will appear stained. If acid coal-tar dyes are used, the liquid contents of the cell will show the color.

Detection of Frozen Meat.—Maljean ‖ detects frozen meat by the aid of a microscope. A drop of the blood or meat juice is pressed out upon a slide and immediately examined before it solidifies. Fresh meat juice contains many red blood corpuscles, floating in a clear colorless serum, and readily apparent. In blood from frozen meat, the red cor-

* Pharm. Central., 1897, 38, p. 884.

† Commercial Organic Analysis, Vol. IV, p. 294.

‡ U. S. Dept. of Agric., Bureau of Chemistry, Bul. 65, p. 16.

§ Zeits. angewand. Mikrosk, 1895, p. 12.

‖ Jour. Pharm. et Chem., 1892, XXV, p. 348.

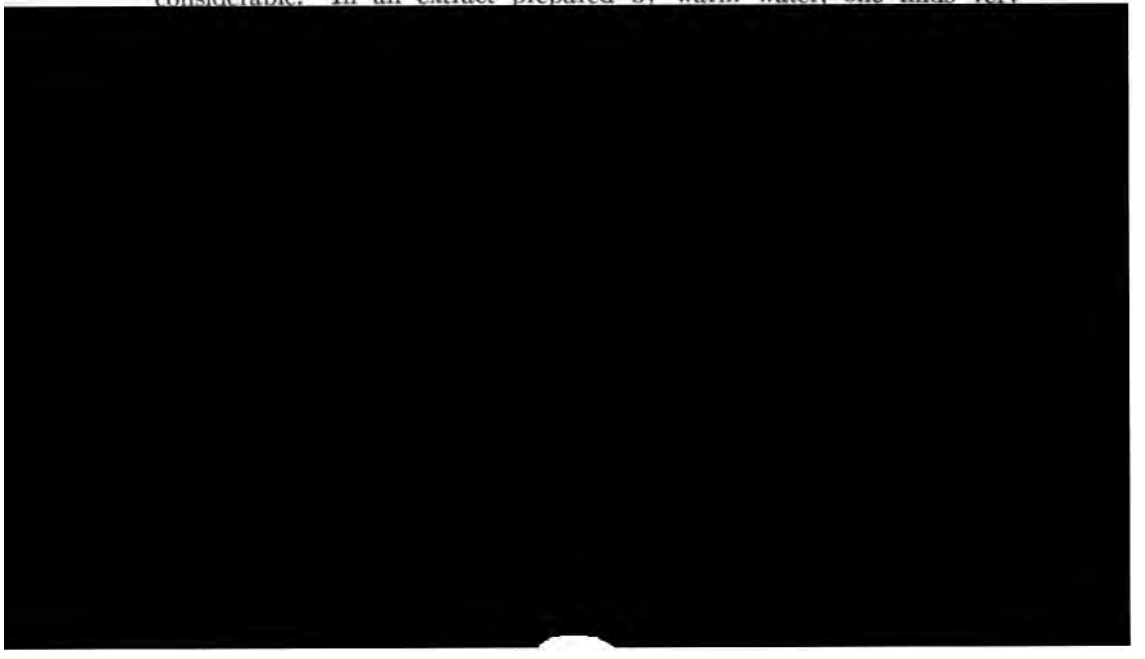
puscles are nearly always completely dissolved in the serum, due to freezing, or, if not dissolved, are much distorted and entirely decolorized, the liquid portion being darker than usual.

Megascopically, the fresh meat juice is more abundant than that of frozen meat, and its color is deeper. According to C. A. Mitchell, if a small piece of meat once frozen be shaken in a test-tube with water, color is imparted to the water much more quickly than with fresh meat, and the color is deeper.

MEAT EXTRACTS.

Character and Composition.—Numerous preparations sold under the name of meat extracts have been on the market for many years. At the beginning of the nineteenth century the value of such extracts was known, but Liebig was the first some fifty years later to produce a commercial extract of meat. Liebig's preparation, as originally made, consisted of a cold-water extract of chopped lean meat, strained free from fiber, heated, filtered, and evaporated, thus containing little of any gelatin or proteids. Later, however, Liebig advocated the use of warm and even boiling water for extraction, by which method of preparation the amount of gelatin was greatly increased. He, however, condemned the use of salt.

The best modern meat extracts consist for the most part of such portions of the meat, previously freed from bone and superfluous fat, as are soluble in water the temperature of which does not exceed 75° C. The widest latitude, however, prevails as to the temperature employed for the extraction, hence the character of the various products is somewhat varied. It is not an uncommon practice to submit the meat to actual boiling with water, in which case the amount of gelatin will be considerable. In an extract prepared by warm water, one finds very



the extent to which evaporation is carried, varying from the thin fluid through the pasty form to the semi-solid. Some preparations have added thereto finely ground dried beef or beef meal.

The following table shows results obtained by Otto Hehner on some of the principal commercial meat extracts:

	Water.	Petroleum-ether Extract.	Gelatin.	Albumin.	Meat Fiber and Coagulable Albumin.	Albumoses.	Peptones.
1. Liebig Company's *Extractum Carnis*	15.26	0.34	5.18	.....	2.12	2.01	8.06
2. Armour's extract of meat.....	15.97	0.21	3.31	.....	.....	1.75	5.13
3. Brand & Co.'s *Extractum Carnis*...	17.85	0.38	4.56	.....	1.81	4.19	10.16
4. Liebig's extract (Bovril Co.).....	22.24	0.29	5.50	.....	1.30	3.62	8.44
5. Brand & Co.'s meat juice.....	55.48	0.10	0.69	1.00	.....	1.06	2.50
6. Valentine's meat juice.....	55.53	0.10	0.75	0.25	.....	2.00	2.87
7. Wyeth's meat juice.....	61.61	0.08	1.12	5.62	.....	1.08	1.86
8. Borthwick's bouillon	36.19	0.25	1.37	.....	4.00	1.16	11.09
9. Vitalia meat juice.....	70.19	0.32	0.45	16.44	0.37	0.05	0.37
10. Brand & Co.'s essence of beef.....	89.69	0.06	5.18	.....	.....	0.19	0.57
11. Bovril Co.'s beef fluid.....	28.34	1.02	3.81	.....	5.37	8.38	13.18

	Meat Bases.	Ash.	Difference.	Sodium Chloride.	Phosphoric Acid.	Total Nitrogen.
1. Liebig Company's *Extractum Carnis*	39.32	23.51	4.20	5.81	6.97	9.07
2. Armour's extract of meat.....	41.12	29.36	3.15	9.74	6.76	8.21
3. Brand & Co.'s *Extractum Carnis*...	38.90	18.80	2.87	3.31	5.16	9.80
4. Liebig's extract (Bovril Co.).....	38.59	20.45	−0.42	5.14	5.50	9.19
5. Brand & Co.'s meat juice.....	12.50	11.06	15.61*	4.43	1.52	2.81
6. Valentine's meat juice.....	12.48	12.01	14.01	2.35	2.85	2.92
7. Wyeth's meat juice	9.44	14.78	4.41	6.96	3.01	3.06
8. Borthwick's bouillon	24.25	17.93	3.76	6.09	3.58	6.70
9. Vitalia meat juice.....	2.82	6.65	2.34	5.11	0.37	3.28
10. Brand & Co.'s essence of beef.....	3.43	1.00	−0.05	0.33	0.40	1.49
11. Bovril Co.'s beef fluid.....	19.38	17.67	2.85	9.07	4.05	8.02

* Glycerin (?).

In calculating the percentage of the various nitrogenous compounds, the factor 6.25 was used in all cases, this being in Hehner's opinion the fairest and best approximation to the true state of affairs in expressing the analysis as a whole, while admitting that this factor is too high in certain cases, as, for instance, in that of the flesh bases.

McGill † gives the following results of the analysis of twenty-three brands of meat extracts:

† Dept. of Inl. Rev. Canada, Bul. 63, p. 14.

OF PASTY CONSISTENCY, CONTAINING FROM 20 TO 40 PER CENT WATER.

er.	Ash.	Chlorine. Calculated to NaCl.	Fat, Petroleum-ether Extract.	Total Nitrogen.	Nitrogen in Portion Insoluble in Water.	Equivalent Proteids, N×6.25.	Nitrogen in Soluble Matter Ppt'd by Bromine.	Equivalent Proteids N×6.25.	Nitrogen in Soluble Matter not Ppt'd by Bromine.	Equivalent Flesh Bases N×3.12.
9	22.2	8.70	1.07	7.630	0.572	3.575	1.008	6.290	6.050	18.876
2	17.9	7.13	1.13	7.100	0.658	4.113	0.475	2.969	5.967	18.617
7	14.0	6.85	0.77	7.660	0.812	5.075	3.010	18.810	3.838	11.975
5	30.0	9.81	0.59	6.853	0.174	1.008	1.029	6.431	5.591	17.444
2	31.4	16.00	0.75	5.381						
8	28.4	19.41	0.52	7.335	0.497	3.106	1.862	11.637	4.976	15.525
5	23.3	11.44	0.14	7.514	0.387	2.419	2.800	17.500	6.327	19.740
9	22.6	9.97	0.37	10.143	0.224	1.400	1.789	11.182	8.130	25.366
8	17.6	3.20	1.02	9.380	0.549	3.431	0.868	5.425	7.963	24.845
2	33.0	21.70	0.23	5.970	0.130	0.813	1.513	9.456	2.842	8.867
	EXTRACTS, MORE OR LESS LIQUID, 40 TO 70 PER CENT WATER.									
3	8.95	6.32	1.23	3.790	1.443	9.019	0.900	5.625	1.447	4.515
3	16.90	11.74	0.12	2.862	0.071	0.444	0.591	3.696	2.200	6.864
4	10.60	7.02	0.42	2.659						
9	13.60	4.66	1.10	3.348	0.412	2.575	0.449	2.806	2.487	7.759
6	11.10	1.45	0.94	3.163	None	.....	0.321	2.006	2.842	8.867
6	14.40	5.49	1.01	4.744	1.058	6.613	1.807	11.294	1.879	5.863
5	13.60	4.78	1.04	5.202	0.849	5.306	1.556	9.725	2.797	8.727
2	16.30	6.63	0.53	3.176	0.093	0.581	0.301	1.881	2.782	8.680
8	16.44	10.91	.....	3.072	0.631	3.944	1.051	6.569	1.390	4.337
	MEAT PREPARATIONS OF EXCEPTIONAL CHARACTER.									
0	1.47	0.84	0.27	2.590	None	.....	2.264	14.150	0.326	1.017
4	0.88	0.33	0.79	0.931	None	.....	0.079	0.793	0.852	3.058
6	1.27	0.19	None	1.475	None	.....	0.868	5.425	0.607	1.894
6	4.60	0.96	0.71	3.840	2.625	16.406	1.246	7.888	None	

METHODS OF ANALYSIS.

Water.—According to Stutzer * water is best estimated by weighing from 5 to 7 grams of the preparation (if of the dry or pasty variety), or from 20 to 25 grams of the fluid extract into a large platinum dish, the dry variety being dissolved in a little hot water. A sufficient amount of previously ignited asbestos or sand, sifted free from dust, is added to absorb the liquid, and the whole is dried to a constant weight in the air-oven.

Ash.—The residue from the above is carefully incinerated in the original dish and weighed. Ammonium nitrate may be employed in burning, if necessary. The chlorine is determined volumetrically or gravimetrically in a solution of the ash.

Fat.—This is best obtained by extracting a portion of the air-dried substance with petroleum ether in a Soxhlet apparatus. Petroleum ether extracts the fat only, while ether extracts other substances as well. A properly prepared extract has very little fat.

Total Nitrogen.—The extract should be tested for nitrates, and the proper modification of the Gunning method should be employed, depending on the presence or absence of nitrates. Use from 1 to 5 grams for the determination.

Separation of Nitrogenous Compounds.—To correctly gauge the food value of a meat extract, it is essential to separate and estimate at least roughly its principal nitrogenous components. To attempt to make such a separation with a high degree of accuracy would involve a long and tedious series of operations, which in most cases would be impracticable. Usually the separation into three main groups, as outlined on page 181, is sufficient. At times, however, it may become necessary, or at least desirable for specific purposes, to determine certain of the nitrogenous compounds separately. Various approximate quick methods are in use for this purpose, and by reason of the fact that in all these methods each class of nitrogenous substances that should theoretically be separated under given conditions is contaminated to some extent with members of other classes, or is itself incompletely separated, it is best to specify in all cases the particular method employed. The most common process among the manufacturing chemists is to employ alcohol of varying strength for fractional precipitation.

Bruylants † adds sufficient alcohol to the solution to amount to 40%

* Zeits. anal. Chemie, 1895, p. 368.

† Jour. Pharm. et Chem., 1897, V, p. 515.

strength. This precipitates mainly the gelatin. The concentrated filtrate from this precipitation is next treated with alcohol to the strength of 80%, which separates out the albumose. Finally the peptones are removed by alcohol of 93 to 94 per cent strength, the flesh bases remaining in solution. Nitrogen is determined separately in each portion.

*Hehner** roughly separated the gelatinous and proteid substances from the flesh bases by the use of methylated spirit (a mixture of ethyl alcohol with 10% of methyl alcohol). He dissolved about 2 grams of the sample in 25 cc. of water, added 50 cc. of strong, methylated spirit, and allowed the mixture to stand over night. He then decanted off the clear supernatant liquid, and dissolved the precipitate in hot water without washing, after which he evaporated the solution to dryness in a tared dish at 100° C. and weighed.

Wiley's Classification † for practical purposes is convenient and very satisfactory. It was used by McGill in obtaining the results given on page 194.

The total nitrogen is divided into three groups as follows:

1st. Nitrogen in the insoluble proteids.

2d. Nitrogen in the soluble proteids, as precipitated with bromine in acid solution.

3d. Amido-nitrogen (including the flesh bases) in the filtrate from the bromine precipitation.

(1) *Insoluble Nitrogen.*—A weighed amount of the sample is treated with a large excess of warm water, the insoluble residue separated by filtration, dried, and weighed. The whole or an aliquot part of this residue is then used for determination of nitrogen by the Gunning method. $N \times 6.25 =$ insoluble protein.



Complete Separation of Nitrogen Compounds would involve a discrimination between meat fiber and insoluble protein, coagulable albumin, acid albumin (syntonin), gelatin, peptones, albumoses, flesh bases, and ammonia.

(1) *Meat Fiber.*—If meat fiber is found by examination with the microscope to be present, about 5 grams of the extract of the dry, or 20 to 25 grams of the fluid variety is exhausted with 200 to 250 cc. water at about 20° C., and the residue collected on a tared filter. It is often difficult to filter such an extract in the ordinary way, and the use of the centrifuge is helpful, passing the clear supernatant liquid through the filter, and finally washing the residue thereon. The residue is washed, dried at 100°, and weighed, or the nitrogen may be determined by the Gunning method. N×6.25 = total insoluble matter, which includes, besides the meat fiber, the insoluble proteids.

(2) *Coagulable Albumin.*—The filtrate from (1) is acidified with acetic acid and boiled for some minutes to coagulate the albumin, which is collected upon a filter (using to advantage a centrifuge as in the preceding paragraph). Determine the nitrogen in the washed residue, using the factor 6.25 for coagulable albumin.

(3) *Acid Albumin or Syntonin.*—Exactly neutralize the filtrate from (2) with weak alkali. If a precipitate occurs, indicating syntonin, it is collected on a filter, washed, and the nitrogen determined. N×6.25 = syntonin.

(4) *Albumoses or Proteoses.*—An aliquot part of the filtrate from (3), or if syntonin be found absent, of the filtrate from (2), is saturated with zinc sulphate, adding the powdered salt as long as it continues to dissolve with stirring and shaking. Proteoses and any traces of gelatin or insoluble proteids that have escaped removal are precipitated, but not the peptones or flesh bases. Filter, wash, and determine the nitrogen in the residue, using the factor 6.25 for the proteoses.

(5) *Peptones.*—The filtrate from (4) or an aliquot part thereof is acidified with hydrochloric acid and treated with bromine in the manner described on page 182. The residue is collected and washed. The nitrogen therein×6.25 = peptones.

(6) *Flesh Bases* are determined by subtracting from the total nitrogen the sum of the nitrogen in (1), (2), (3), (4), and (5), and multiplying the remainder by the factor 3.12.

Two samples of commercial meat extract were analyzed by A. McGill by methods closely analogous to the above, with the following results:

	Vigoral.	Vimbos.
Insoluble residue	8.95	6.32
Total nitrogen	1.047	0.906
Equivalent protein (N×6.25)	6.544	5.663
Soluble matter	47.45	50.18
Nitrogen in coagulable albumins	0.0196	0.058
Coagulable albumin (N×6.25)	0.1225	0.363
Syntonin (acid albumin)	None	None
Proteose nitrogen (by zinc sulphate)	0.314	0.391
Equivalent proteoses (N×6.25)	1.963	2.444
Peptone nitrogen	1.059	1.400
Equivalent peptones (N×6.25)	6.621	8.750
Amido-nitrogen (flesh bases) by dif.	2.305	2.447
Equivalent flesh bases (N×3.12)	7.192	7.635

Determination of Gelatin. — This is accomplished by the modified Stutzer method as given on page 183.

Determination of Ammoniacal Nitrogen.—This is best estimated, according to Stutzer, by dissolving 5 to 25 grams of the preparation in water (if the extract is not of the fluid variety), adding barium carbonate, and distilling into a standardized acid. Nitrogen in the form of ammonia salts is rarely present in these preparations.

PRESERVATIVES IN MEAT EXTRACTS.—Boric acid is sometimes used as a preservative in these preparations and is tested for by the usual methods (p. 185).

FISH.

Structure and Composition.—Fish resembles meat both structurally and in the nature of its constituents, but differs from it in a marked degree in the relative proportions of its various components. Thus, there is considerably more refuse matter such as skin and bones in fish than in meat, and in the edible portion of fish the amount of water is much greater.



Water	740.82
Albumin	137.40
Collagen	43.88
Fat	45.97
Extractives	16.97
Salts	14.96

Hutchison classifies fish as follows with reference to their content of fat:

Lean.—Fish having less than 2% fat, such as cod and haddock.

Medium.—Fish having 2 to 5% fat, such as halibut and mackerel.

Fat.—Fish having more than 5% of fat, such as eel, 18%; salmon, 12%; turbot, 12%, and herring, 8%.

According to Atwater and Bryant * the composition of different varieties of fish is as follows:

COMPOSITION OF FISH.

		Refuse.	Water.	Protein.		Fat.	Ash.	Fuel Value per Pound.
				N × 6.25.	By Difference.			
Bass—	edible portion	.....	77.7	18.6	18.3	2.8	1.2	465
	as purchased	55.0	35.1	8.4	8.3	1.1	0.5	200
Bluefish—	edible portion	.....	78.5	19.4	19.0	1.2	1.3	410
	as purchased	48.6	40.3	10.0	9.8	0.6	0.7	210
Cod—	edible portion	.....	82.6	16.5	15.8	0.4	1.2	325
	as purchased	52.5	38.7	8.4	8.0	0.2	0.6	165
Eel—	edible portion	.....	71.6	18.6	18.3	9.1	1.0	730
	as purchased	20.2	57.2	14.8	14.6	7.2	0.8	580
Haddock—	edible portion	.....	81.7	17.2	16.8	0.3	1.2	335
	as purchased	51.0	40.0	8.4	8.2	0.2	0.6	165
Halibut—	edible portion	.....	75.4	18.6	18.4	5.2	1.0	565
	as purchased	17.7	61.9	15.3	15.1	4.4	0.9	470
Herring—	edible portion	.....	72.5	19.5	18.9	7.1	1.5	660
	as purchased	42.6	41.7	11.2	10.9	3.9	0.9	375
Mackerel—	edible portion	.....	73.4	18.7	18.3	7.1	1.2	645
	as purchased	44.7	40.4	10.2	10.0	4.2	0.7	365
Perch—	edible portion	.....	75.7	19.3	19.1	4.0	1.2	530
	as purchased	62.5	28.4	7.3	7.2	1.5	0.4	200
Pickerel—	edible portion	.....	79.8	18.7	18.6	0.5	1.1	370
	as purchased	47.1	42.2	9.9	10.7	0.3	0.6	210
Salmon—	edible portion	.....	64.6	22.0	21.2	12.8	1.4	950
	as purchased	34.9	40.9	15.3	14.4	8.9	0.9	660
Shad—	edible portion	.....	70.6	18.8	18.6	9.5	1.3	750
	as purchased	50.1	35.2	9.4	9.2	4.8	0.7	380
Skate—	edible portion	.....	82.2	18.2	15.3	1.6	1.1	400
	as purchased	51.0	40.2	8.9	7.5	0.7	0.6	195
Smelt—	edible portion	.....	79.2	17.6	17.3	1.8	1.7	405
	as purchased	41.9	46.1	10.1	10.0	1.0	1.0	230
Trout—	edible portion	.....	77.8	19.2	18.9	2.1	1.2	445
	as purchased	48.1	40.4	9.9	9.8	1.1	0.6	230
Turbot—	edible portion	.....	71.4	14.8	12.9	14.4	1.3	885
	as purchased	47.7	37.3	7.7	6.8	7.5	0.7	460
Whitefish—	edible portion	.....	69.8	22.9	22.1	6.5	1.6	700
	as purchased	53.5	32.5	10.6	10.3	3.0	0.7	325

* U. S. Dept. of Agric., Off. of Exp. Sta., Bul. 28, p. 47 et seq.

Crustaceans and Mollusks.—These differ from the meats and common fish by reason of the presence in considerable proportion of the carbohydrate glycogen, contained in the liver. The *lobster* and *crab* are nearly alike in composition, the flesh being made up of coarse, dense, thick-walled fibers.

Payen gives the following composition of the flesh and body of lobster:

	Flesh (contained in Claws and Tail).	Body (consisting mainly of Liver).
Water	76.6	84.31
Proteid	19.17	12.14
Fat	1.17	1.14

Clams and *Oysters* are low in solid nutriment, and are more digestible when eaten raw than cooked. Oysters contain 3% or more of glycogen.

The following analyses are from Atwater and Bryant:*

COMPOSITION OF SHELL FISH, ETC.

		Refuse.	Water.	Protein. N × 6.25.	Fat.	Carbohydrates.	Ash.	Fuel Value per Pound. Cals.
Clams—	edible portion	.....	85.8	8.6	1.0	2.0	2.6	240
	as purchased	41.9	49.9	5.0	0.6	1.1	1.5	140
Crabs—	edible portion	.....	77.1	16.6	2.0	1.2	3.1	415
	as purchased	52.4	36.7	7.9	0.9	0.6	1.5	195
Lobster—	edible portion	.....	79.2	16.4	1.8	0.4	2.2	390
	as purchased	61.7	30.7	5.9	0.7	0.2	0.8	140
Mussels—	edible portion	.....	84.2	8.7	1.1	4.1	1.9	285
	as purchased	46.7	44.9	4.6	0.6	2.2	1.0	150
Oysters—	edible portion	.....	86.9	6.2	1.2	3.7	2.0	235
	as purchased	81.4	16.1	1.2	0.2	0.7	0.4	45
Scallops—	as purchased	.....	80.3	14.8	0.1	3.4	1.4	345
Terrapin—	edible portion	.....	74.5	21.2	3.5	.....	1.0	545
	as purchased	75.4	18.3	5.2	0.9	.....	0.2	135

gills, or that possesses the marked odor that accompanies incipient decomposition, should not be used as food.

Methods of Analysis.—These are similar to the methods given for meat.

Preservatives in Fish and Oysters.—Boric acid and borax in mixture form the most common preservatives of salt dried fish and of oysters. In the case of salt codfish, the boric mixture is sprinkled on the surface. Such surface application in some states, as for example Massachusetts, is allowed by law. In opened oysters sold in casks and kegs, the liberal use of boric mixture is frequent in solution in the oyster liquor.

The author has found salicylic acid in bottled clam juice and clam bouillon.

CONCENTRATED FOODS.

Under the name of "condensed" or "concentrated foods" or "emergency rations" a number of canned preparations are sold for the use of campers, travelers, armies in the field, etc. These consist usually of mixtures of dried ground meats and vegetables, pressed together in compact form, and preserved in tin cans. The claims made for the food value of these preparations are, as a rule, extravagant and erroneous, as shown by Woods and Merrill,* who give the following analyses of some of these foods:

	Net Weight Contents.	Weight of Materials in Package.					Total Fuel Value. Cals.
		Water.	Proteids.	Fat.	Carbohydrates.	Ash.	
	Grams.	Grams.	Grams.	Grams.	Grams.	Grams.	
Ration cartridge, pea, beef, etc.	241	34.2	52.9	42.0	98.0	13.9	1071
Blue ration campaigning food, *a*	169	76.1	37.5	9.0	37.9	8.5	432
" " " " *b*	78	1.0	5.6	23.1	46.9	1.4	436
Red ration campaigning food, *a*	122	33.8	26.2	18.5	37.8	5.7	496
" " " " *b*	77	1.2	5.0	23.0	46.6	1.2	424
Ration cartridge, potatoes, beef, etc.	283	117.9	62.3	12.6	76.4	13.8	772
Emergency ration, *a*	120	14.2	56.1	29.6	11.9	7.8	617
" " *b*	113	1.9	8.2	32.7	68.0	2.2	622
Emergency ration, *a*	121	4.5	71.8	32.6	6.7	5.4	776
" " *b*	127	5.7	8.3	15.3	94.8	2.9	588
Nao meat food	437	231.3	56.9	90.1	46.2	12.5	1328
Army rations	661	420.2	101.2	84.3	47.9	7.4	1542
Standard emergency ration	418	23.6	129.6	90.5	160.3	14.0	2198
" " " *a*	270	17.0	50.6	54.8	137.0	10.6	1402
" " " *b*	49	0.5	3.2	10.5	34.0	0.8	254
Arctic food	423	30.7	75.1	167.3	119.8	30.1	2430
Tanty emergency ration	475	313.5	60.2	48.6	41.9	10.8	1482
F-A Food Company's stew	964	638.0	149.2	114.5	52.5	9.8	2460

* Maine Exp. Sta., Bul. 75, p. 103.

REFERENCES ON FLESH FOODS.

ANDREWS, O. W. Flesh Foods. London, 1900.
ATKEN, W. On the Animal Alkaloids. London, 1890.
BAILLET, L. Traité de l'Inspection des Viandes.
BALLAND, A. The Composition of Fish, Crustaceans and Molluscs. Compt. rend., 1898, 126, 1721.
BIGELOW, W. D. Meat and Meat Products. U. S. Dept. of Agric., Bureau of Chemistry, Bul. 65, p. 7.
BRIEGER, L. Untersuchung ueber Ptomaine. Berlin, 1885.
COBBOLD, T. S. Internal Parasites of Domestic Animals.
—— Parasites of Man.
—— Entozoa of Man and Animals. London, 1865.
DENAEYER. La Composition des Peptones de Viande, 1896.
DOUGLAS'S Encyclopædia for Bacon Curers, Meat Inspectors, Local Authority Officers, etc. Wm. Douglas & Sons, London.
DUFF, JAS. C. Manufacture of Sausages. New York, 1899.
FALCK, C. Das Fleisch. Marburg, 1880.
FISCHODER, F. Leitfaden der Praktischen Fleischbeshau. Berlin, 1899.
GAUTIER, A. Les Toxines. Paris, 1896.
GRINDLEY, H. S. Losses in Cooking Meat. Exp. Sta. Bul. 102.
GUENTHER. The Study of Fishes.
HOFMANN. Lehrbuch der Zoochemie.
LANGWORTHY, C. F. Fish as Food. Farmer's Bul. 85.
LEBBEN, S. Preservation and Coloring of Meat Produce. Berlin, 1901.
LEUCKART. Human Parasites.
MALLET, J. W. Physiological Effect of Creatine and Creatinine, and their Value as Nutrients. U. S. Dept. of Agric., Off. of Exp. Sta., Bul. 66.
MITCHELL, C. A. Flesh Foods. London, 1900.
MCGILL, A. Commercial Beef Extracts. Canada Inland Rev. Dept., Bul. 63.
OSTERTAG. Handbuch der Fleischbeshau.
SALMON, D. E. Inspection of Meats for Animal Parasites. Bul. 19, Bureau of An.



CHAPTER VIII.

EGGS.

Nature and Composition.—Though eggs of various birds are used to some extent as food, it is the egg of the hen that is in universal use for this purpose, and therefore the one which is here for the most part discussed, bearing in mind that the structure and composition of all varieties of birds' eggs are closely analogous.

Fig. 55 shows the longitudinal section of a hen's egg.

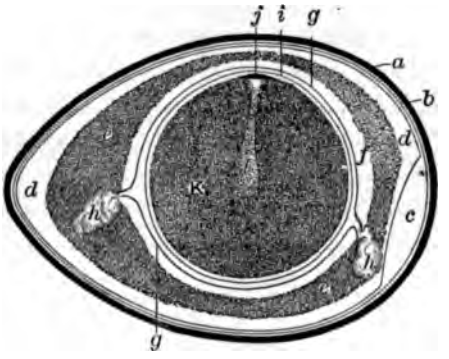


FIG. 55.—Longitudinal Section of a Hen's Egg. *a*, Shell; *b*, Double Membrane of Shell; *c*, Air-chamber; *d*, Outer, or Fluid Albuminous Layer; *e*, Thick, Middle Albuminous Layer; *f*, Inner Albuminous Layer; *g*, Membrane of the Chalaza; *hh*, the Chalaza; *i*, Vitelline Membrane; *j*, Germ; *k*, Yolk; *l*, Latebra. (After Macé.)

The average weight of a hen's egg is 60 grams, of which the shell weighs about 6, the white 36, and the yolk 18. Roughly it contains 70% of water, 12% of albumin, and 12% of fat.

The shell, according to König, has the following composition:

Calcium carbonate	89–97%
Magnesium carbonate	0– 2%
Calcium and magnesium phosphate	0.5– 5%
Organic substances	2.0– 5%

The mean percentage composition of the eggs of the hen, duck, and plover are, according to König, as follows:

	Water, Per Cent.	Proteids, Per Cent.	Fat, Per Cent.	Nitrogen-free Substance Per Cent.	Salts Per Cent.	In the Dry Substance.	
						Nitrogen Per Cent.	Fat Per Cent.
Hen's egg	73.67	12.55	12.11	0.55	1.12	7.66	45.99
Duck's egg	71.11	12.24	15.49	....	1.16	6.78	53.62
Plover's egg	74.43	10.75	11.66	2.18	0.98	6.75	45.78
White of hen's egg	85.75	12.67	0.25	....	0.59	14.25	1.78
Yolk " " "	50.79	16.24	31.75	0.13	1.09	5.30	64.43

The Egg-white.—The white of egg has a specific gravity of 1.045, and its reaction is always alkaline. It is a transparent, albuminous fluid inclosed in a framework of thin membrane. The fibrous portion of the membrane is insoluble in water and in dilute acetic acid.

The composition of the fluid substance of the white of egg, according to Lehmann, is as follows:

Water	82 to 88%
Solids	13.3% (mean)
Proteids	12.2% "
Sugar	0.5% "
Fats, alkaline soaps, lecithin, cholesterin	traces
Inorganic residue	0.66%

The proteid substance is for the most part albumin, with a small amount of globulin.

According to Osborne and Campbell * the nitrogen compounds of the white of egg are four in number, which they name ovalbumin, ovo-



in dilute salt solution at a lower temperature (below 60°), and the coagulum is more flocculent than that of ovalbumin.

Ovomucoid is not coagulable by heat, and may thus be separated (imperfectly) by filtering out all the coagulable proteids.

The last two compounds exist in very small amounts only.

Preparation of Albumin.*—By beating up the white of egg in water, the salts and the albumin are dissolved, while the fibrous portion is insoluble and is removed by filtration. The filtrate is then treated with a slight excess of basic lead acetate, the precipitate decomposed by treatment with carbon dioxide, and the lead removed by hydrogen sulphide. The solution is warmed cautiously to 60° C., thus beginning to coagulate the albumin, a small part of which, coming down in a flaky form, carries with it the lead sulphide. On filtering or pouring off the supernatant liquid after cooling, one obtains a colorless solution of the albumin, which is evaporated to dryness below 40°. The albumin is obtained in the form of transparent yellowish, horny scales, which may be pulverized in a mortar, if desired. Its specific gravity is 1.262. It is tasteless, odorless, and neutral in reaction, and slowly soluble in water.

The Egg-yolk.—This is much more complex in composition than the white. Halliburton thus enumerates the constituents of the yolk:

(*a*) *Proteids.*—Vitellin, the chief one, a globulin resembling myosin.

Albumin, in small quantities.

Nuclein, combined chiefly with the iron present.

(*b*) *Fats.*—Olein, palmitin, and stearin.

A yellow lipochrome or lutein.

(*c*) *Carbohydrates.*—Grape sugar in small quantities.

(*d*) *Other Organic Constituents.* — Lecithin, a phosphorized nitrogenous body allied both to the fats and to the proteids.

Cerebrin.

Cholesterin.

(*e*) *Inorganic Salts*, the most abundant of which is potassium chloride. Gobley gives the following composition to the egg-yolk:

	Per Cent.		Per Cent.
Vitellin	15.8	Cholesterin	0.4
Nuclein	1.5	Fats	20.3
Cerebrin	0.3	Coloring matters	0.5
Lecithin	7.2	Salts	1.0
Glycerol phosphoric acid	1.2	Water	51.8

* Allen, Com. Org. Anal., Vol. IV, p. 42.

Osborne and Campbell,* as the result of long and careful experiments, consider the protein of egg-yolk to be largely if not wholly a lecithin compound, having properties of a globulin, and soluble in sodium chloride solution.

The fat of the egg yolk, which is used in ointments, has the following characteristics according to Spaeth:†

Specific gravity at 100° C.	0.881
Iodine number	68.48
Reichert-Meissl value	0.66
Refractive index at 25° C. (on butyro-refractometer scale)	68.5
Melting-points of fatty acids	36° C.
Iodine number of fatty acids	72.6

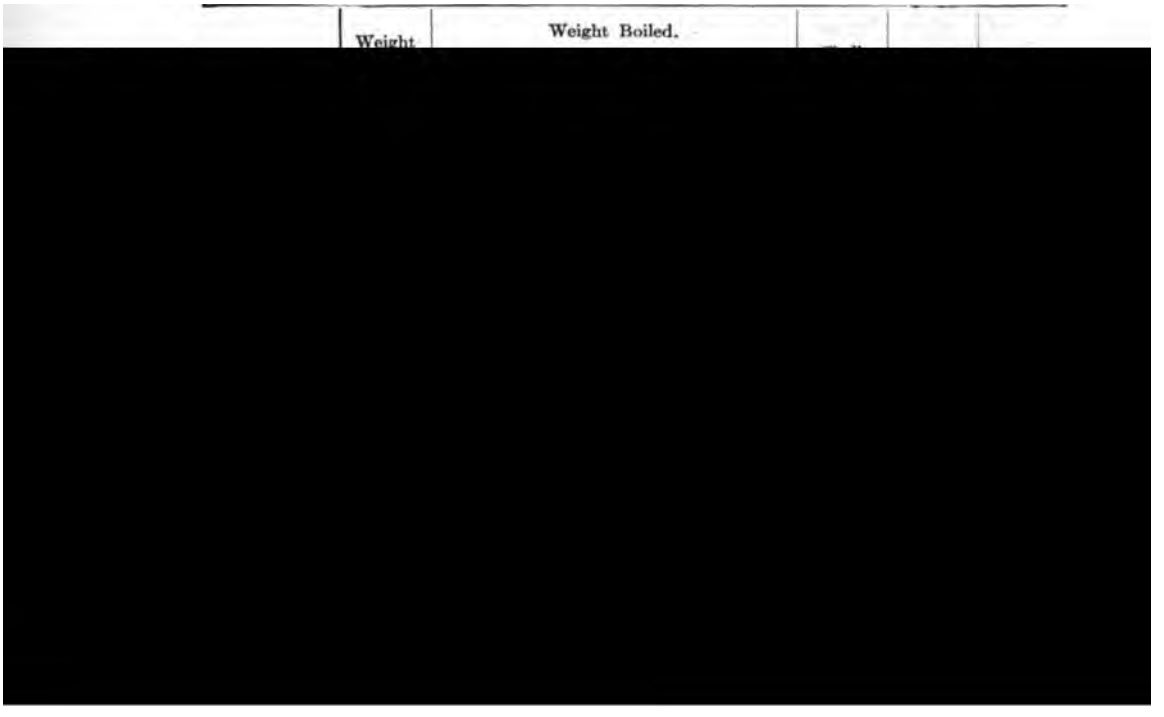
The mineral content of the egg is thus shown by König:

COMPOSITION OF THE ASH OF EGGS.

	Ash of the Dry Substance.	Potash.	Soda.	Lime.	Magnesia.	Iron Oxide.	Phosphoric Acid.	Sulphuric Acid.	Silica.	Chlorine.
Hen's egg: entire..	3.48	17.37	22.87	10.91	1.14	0.39	37.62	0.32	0.31	8.98
white..	4.61	31.41	31.57	2.78	2.79	0.57	4.41	2.12	1.06	28.82
yolk...	2.91	9.29	5.87	13.04	2.13	1.65	65.46	.....	0.86	1.95

The following analyses of eggs were made by Wood and Merrill:‡

AVERAGE WEIGHTS OF EGGS AND PARTS AS PREPARED FOR ANALYSIS.



COMPOSITION OF EGGS.

		Refuse (Shells).	Water.	Protein.		Fat.	Ash.	Fuel Value per Pound.
				Nitrogen ×6.25.	By Difference.			
								Cal.
Turkey—	white	.....	86.7	11.5	12.5	Trace	0.8	325
	yolk	.....	48.3	17.4	17.6	32.9	1.2	1875
	entire edible portion	.....	73.3	13.4	14.2	11.2	0.9	850
	as purchased	13.8	63.5	11.6	12.2	9.7	0.8	735
Goose—	white	.....	86.3	11.6	12.9	Trace	0.8	330
	yolk	.....	44.1	17.3	18.4	36.2	1.3	1975
	entire edible portion	.....	69.5	13.8	15.1	14.4	1.0	985
	as purchased	14.2	59.7	11.5	12.9	12.3	0.9	860
Duck—	white	.....	87.0	11.1	12.2	Trace	0.8	315
	yolk	.....	45.8	16.8	16.8	36.2	1.2	1980
	entire edible portion	.....	70.5	13.3	14.0	14.5	1.0	985
	as purchased	13.7	60.9	11.5	12.1	12.5	0.8	880
Guinea fowl—	white	.....	86.6	11.6	12.6	Trace	0.8	325
	yolk	.....	49.7	16.7	17.3	31.8	1.2	1800
	entire edible portion	.....	72.8	13.5	14.3	12.0	0.9	875
	as purchased	16.9	60.6	11.2	11.9	9.9	0.7	730
Hen—	white	.....	86.2	12.3	13.0	0.2	0.6	
	yolk	.....	49.5	15.7	16.1	33.3	1.1	
	entire edible portion	.....	73.7	13.4	14.8	10.5	1.0	
	as purchased	11.2	65.5	11.9	13.1	9.3	0.9	

METHODS OF ANALYSIS.

Preparation of the Sample.*—The egg is first weighed as a whole and afterwards boiled hard, cooled, and again weighed. The shell, white, and yolk are then carefully separated and each weighed. After rejecting the shell, the yolk and white are separately reduced by a chopping-knife to the size of wheat grains. These portions are dried partially at a temperature not exceeding 45°, weighed, and afterwards ground to a fine powder in a mortar.

Determinations of water, fat, ash, and total nitrogen are made in practically the same manner as with flesh foods.

Little attention has been paid as yet to the complete separation and determination of the nitrogen compounds in the white and yolk, and it is customary in most cases to express the protein of the whole as N×6.25.

Determination of Lecithin.—*Wiley's Method.*†—The whole egg, excluding the shell, is placed in a flask with a reflux condenser, and boiled for six hours with absolute alcohol. The alcohol is then evaporated off, and the residue treated in like manner for ten hours with ether. After evaporat-

* Woods and Merrill, Maine Exp Sta., Bul. 75, p. 92.

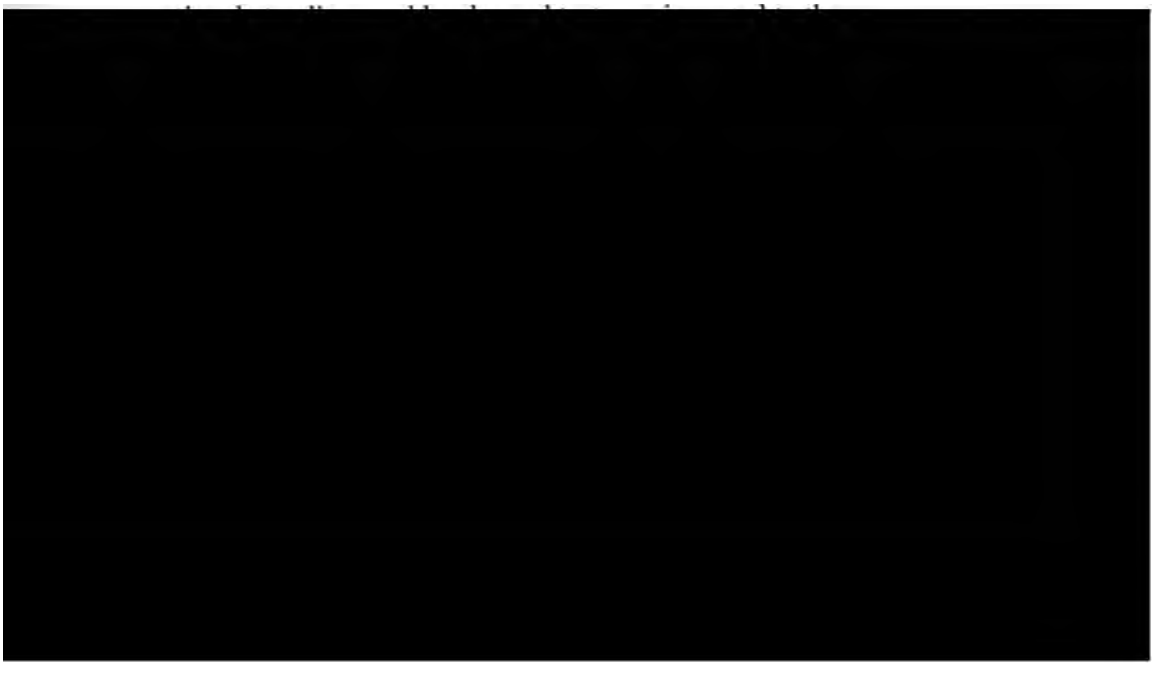
† Principles and Practice of Agricultural Analysis, Vol. III, p. 431.

ing off the ether, the dry residue is rubbed to a fine powder, placed in an extractor and treated with pure ether for ten hours. The ether extract thus secured is oxidized, after removal of the ether, by fusion with mixed sodium and potassium carbonates, and the phosphorus is determined in the usual way as magnesium pyrophosphate. The amount of lecithin is obtained by multiplying the weight of magnesium pyrophosphate by the factor 7.2703, on the basis of Hoppe-Seyler's formula for lecithin: $C_{44}H_{90}NPO_9$.

If, for example, an amount of organic phosphorus yielding 0.0848 gram of magnesium pyrophosphate is found in 54 grams of egg exclusive of shell, then $0.0848 \times 7.2703 = 0.61652$ and $0.61652 \times 100 \div 54 = 1.14$. Therefore the percentage of lecithin in the egg is 1.14.

Preservation of Eggs.—Owing to the porous nature of the shell, the moisture of the contents gradually grows less by evaporation, and the egg loses in weight. Air also passes in through the shell pores, carrying various microbes, which result in ultimate decomposition and spoiling of the egg. Nature has provided the shell with a thin surface coating of mucilaginous matter, which, however, is easily washed off. This coating tends to partially close the pores, and for best results in keeping should not be removed by washing.

Eggs are commonly preserved by protecting them as far as possible from the air. This is accomplished in a variety of ways, the most common being to pack the eggs in salt or bran, so that the packing medium fills up the interstices between the eggs. Eggs thus packed will keep considerably longer then when exposed to the air. A solution of salt is sometimes employed, and also lime water, the eggs being simply packed in the solution. The use of lime water is, however, open to the serious objec-



One drawback to this method is that eggs so treated break more easily on boiling, but this may be prevented by carefully piercing the shell with a strong needle.

Cadet de Vanx has proposed immersing the egg in boiling water for twenty seconds, the result being that a very thin layer of the egg-white next the shell becomes coagulated, thus forming an impervious coating inside the shell.

Physical Examination of Eggs.—Various physical tests have been prescribed for ascertaining the approximate age of an egg. Thus, according to Delarne, if the egg, when placed in a 10% salt solution, sinks to the bottom, it may be considered perfectly fresh; if it remains immersed in the liquid, it is to be considered at least three days old; and if it rises to the surface and floats thereon it is more than five days old. This test is a very rough one, and is useful only for eggs that have been kept in the air. Preserved eggs cannot be gauged by this means.

The best method of examining eggs for freshness consists in interposing the egg between a bright light and the eye. If the egg is fresh, it will show a perfectly uniform rose-colored tint, without dark spots, the air-chamber being small and occupying about one-twentieth the capacity of the egg. If the egg is not fresh, it will appear more or less cloudy, being darker as the egg grows older, becoming in extreme cases opaque. At the same time the air-chamber grows larger as the age increases.

Desiccated Egg.—It is possible to evaporate to dryness the contents of the egg to form a powder, the keeping qualities of which far exceed that of ordinary eggs, while it forms a concentrated food which lends itself much more readily to transportation than does the fresh egg in the shell. Several brands of desiccated egg are on the market, which from their analyses are undoubtedly genuine. The following are analyses of two of them, one (A) made by the Bureau of Chemistry, the other (B) by the Massachusetts State Board of Health:

	A.	B.
Water	6.80	5.95
Protein (N×6.25)	45.20	48.15
Protein by difference	51.20	
Fat	38.5	40.56
Ash	3.5	5.34

Egg Substitutes.—There have been many preparations in powdered form sold under this name, nearly all claiming to contain all the ingredients

of eggs, but most of them falling far short of these claims. Some of them, as for instance those made from desiccated skimmed milk, do contain nitrogenous matter, but as a rule little if any fat.

Two samples of "egg substitute" sold in Massachusetts were analyzed with the following results:*

	A.	B.
Protein	16.94	18.72
Fat	3.43	3.40
Water	6.71	7.01
Corn-starch, salts, and coloring matter	72.92	70.87

A ten-cent package of sample A, weighing about 2 ounces, was alleged to be equivalent to 12 eggs. Starch furnished the chief ingredient in both samples.

One of the most flagrant examples of fraud in this connection was a product sold under the name "N'egg," advertised to contain the nutritive equivalent of the whites and yolks of a dozen eggs, "their composition being based on careful scientific analysis of natural eggs." It was put up in two small boxes, one containing a white and the other a yellow dry powder. Both were entirely devoid of nitrogen, and consisted of nearly pure tapioca starch with a little common salt, the color of the "yolk" being due to Victoria yellow.

Some egg substitutes are sold under the name of "custard powders," and are alleged to take the place of eggs in cooking. These are variously made up of mixtures of skim-milk powder, coloring matter, and baking powder ingredients as shown from the following analyses:†



REFERENCES ON EGGS.

LANGWORTHY, C. F. Eggs and their Uses as Food. Farmer's Bul. 128.
OSBORNE, T. B., and CAMPBELL, G. F. Proteids of the Egg Yolk. Jour. Am. Chem. Soc., XXII, page 413.
—— Protein Constituents of Egg White. Jour. Am. Chem. Soc., XXII, page 422.
SNYDER, H. Digestibility of Potatoes and Eggs. Exp. Sta. Bul. 43, page 20.

Farmer's Bul. 87. Food Value of Eggs, page 24.
" " 103. Preserving Eggs.

CHAPTER IX.

CEREALS AND THEIR PRODUCTS, LEGUMES, VEGETABLES, AND FRUITS.

THE chief points of difference in composition between the animal foods already treated of, and those of the vegetable kingdom, are apparent in the relative amounts of proteids and carbohydrates. The proteids present in the cereals and vegetables differ materially both in character and amount from those in the flesh foods, being as a rule present to a much greater extent in the meats than in the grains and vegetables. The leguminous foods, such as peas, beans, and lentils, are, however somewhat exceptional in this respect, being comparatively high in nitrogenous content.

The carbohydrates, which in the flesh foods are almost entirely lacking, and in milk and its products are present to a small extent only, form the most important and abundant class of constituents in the vegetable foods.

The composition of the principal cereal grains is tabulated as follows by Villier and Collin:



CEREAL GRAINS.

	Number of Analyses.	Weight of 100 Kernels, Grams.	Moisture.	Proteids.	Ether Extract.	Crude Fiber.	Ash.	Carbohydrates, other than Crude Fiber.	Wet Gluten.	Dry Gluten.
Barley:	14									
Mean	.....	4.533	6.47	11.52	2.67	3.81	2.87	72.66		
Buckwheat:	10									
Mean	.....	3.069	12.31	10.86	2.06	10.57	1.85	63.34		
Corn, domestic:										
Maximum	.....	48.312	12.32	11.55	5.06	2.00	1.55	75.07		
Minimum	.....	10.608	9.58	8.58	2.94	1.00	1.19	68.97		
Mean	.....	38.979	10.93	9.88	4.17	1.71	1.36	71.95		
Oats, domestic:										
Maximum	.....	3.891	13.02	15.05	6.14	16.65	4.37	61.44		
Minimum	.....	2.038	7.87	9.10	0.93	8.57	.47	53.70		
Mean	.....	2.918	10.06	12.15	4.33	12.07	3.46	58.75		
Rice:										
Unhulled	 4	2.929	10.28	7.95	1.65	10.42	4.09	65.60		
Unpolished	 6	2.466	11.88	8.02	1.96	0.93	1.15	76.05		
Polished	 14	2.132	12.34	7.18	0.26	0.40	0.46	79.36		
Rye, domestic:										
Maximum	.....	4.201	11.45	18.99	2.30	2.50	2.41	75.36		
Minimum	.....	1.932	9.54	8.40	1.16	1.65	1.71	63.61		
Mean	.....	2.493	10.62	12.43	1.65	2.09	1.92	71.37		
Wheat, domestic:										
Maximum	.....	6.190	14.53	17.15	2.50	3.72	2.35	76.05	39.05	14.65
Minimum.	.....	2.125	7.11	8.58	0.28	1.70	1.40	66.67	12.33	4.70
Mean	.....	3.866	10.62	12.23	1.77	2.36	1.82	71.18	26.46	10.31
Wheat, foreign:										
Maximum	.....	5.723	12.97	14.52	2.26	2.89	2.04	76.14	32.57	12.33
Minimum.	.....	2.250	8.52	8.58	0.73	1.87	1.67	67.01	18.72	7.00
Mean	.....	4.076	11.47	12.08	1.78	2.28	1.73	70.66	25.36	9.82

Balland * gives the following percentage composition of beans, lentils, and peas:

	Beans.		Lentils.		Peas.	
	Min.	Max.	Min.	Max.	Min.	Max.
Water	10.10	20.40	11.70	13.50	10.60	14.20
Nitrogenous substances	13.81	25.46	20.42	24.24	18.88	22.48
Fat	0.98	2.46	0.58	1.45	1.22	1.40
Sugars and starches	52.01	60.98	56.07	62.45	56.21	61.10
Cellulose	2.46	4.62	2.96	3.56	2.90	5.52
Ash	2.38	4.20	1.99	2.66	2.26	3.50

* Jour. Pharm. Chem., 1897, pp. 196, 197.

The composition of potatoes, according to Balland,* is as follows:

		Water.	Nitrogenous Substances.	Fat.	Sugar and Starches.	Cellulose.	Ash.
Normal state—	minimum ..	66.10	1.43	0.04	15.58	0.37	0.44
	maximum. .	80.60	2.81	0.14	29.85	0.68	1.18
Dried—	minimum ..	.....	5.98	0.18	80.28	1.40	1.66
	maximum. .	.....	13.24	0.56	89.78	3.06	4.38

The composition of the common vegetables, fruits, and berries is thus given by Atwater and Bryant.†

VEGETABLES.

		Number of Analyses.	Refuse.	Water.	Protein.	Fat.	Total Carbohydrates.	Crude Fiber.	Ash.	Fuel Value per Pound, Calories.
Asparagus—	as purchased...	3	.....	94.0	1.8	0.2	3.3	.8	.7	105
Beans, dried—	as purchased...	11	.....	12.6	22.5	1.8	59.6	4.4	3.5	1605
Beans, fresh Lima—	edible portion..	1	.....	68.5	7.1	.7	22.0	1.7	1.7	570
	as purchased...	.....	55.0	30.8	3.2	.3	9.9	.8	.8	255
Beets, fresh—	edible portion..	24	.....	87.5	1.6	.1	9.7	.9	1.1	215
	as purchased...	.....	20.0	70.0	1.3	.1	7.7	.....	.9	170
Cabbage—	edible portion..	16	.....	91.5	1.6	.3	5.6	1.1	1.0	145
	as purchased...	.....	15.0	77.7	1.4	.2	4.8	.....	.9	125
Carrot, fresh—	edible portion..	18	.....	88.2	1.1	.4	9.3	1.1	1.0	210
	as purchased...	.....	20.0	70.6	.9	.2	7.4	.....	.9	160
Celery—	edible portion..	5	.....	94.5	1.1	.1	3.3	.....	1.0	85
	as purchased...	.....	20.0	75.6	.9	.1	2.6	.....	.8	70
Cauliflower—	as purchased...	2	.....	92.3	1.8	.5	4.7	1.0	.7	140
Cucumber—	edible portion..	4	.....	95.4	.8	.2	3.1	.....	.5	80
	as purchased...	.....	15.0	81.1	.7	.2	2.6	.....	.4	70
Lettuce—	edible portion..	8	.....	94.7	1.2	.3	2.9	.7	.9	90
	as purchased...	.....	15.0	80.5	1.0	.2	2.5	.....	.8	75
Mushrooms—	as purchased...	11	.....	88.1	3.5	.4	6.8	.8	1.2	210
Onion, fresh—	edible portion..	15	.....	87.6	1.6	.3	9.9	.8	.6	225

FRUITS.

		Number of Analyses.	Refuse.	Water.	Protein.	Fat.	Total Carbohydrates.	Crude Fiber.	Ash.	Fuel Value per Pound, Calories.
Apples—	edible portion	29	.....	84.6	.4	.5	14.2	1.2	.3	290
	as purchased.....	.....	25.0	63.3	.3	.3	10.8	.....	.3	220
Apricots—	edible portion.....	11	.....	85.0	1.1	.....	13.4	.....	.5	270
	as purchased.....	.....	6.0	79.9	1.0	.....	12.6	.....	.5	255
Bananas—	edible portion.....	6	.....	75.3	1.3	.6	22.0	1.6	.8	460
	as purchased.....	.....	35.0	48.9	.8	.4	14.3	.....	.6	300
Blackberries—	as purchased.....	9	.....	86.3	1.3	1.0	10.9	2.5	.5	270
Cherries—	edible portion.....	16	.....	80.9	1.0	.8	16.7	.2	.6	365
	as purchased.....	.....	5.0	76.8	.9	.8	15.9	.....	.6	345
Cranberries—	as purchased.....	3	.....	88.9	.4	.6	9.9	1.5	.2	215
Currants—	as purchased.....	1	.....	85.0	1.5	.....	12.8	.....	.7	265
Figs, fresh—	as purchased.....	28	.....	79.1	1.5	.....	18.8	.....	.6	380
Grapes—	edible portion.....	5	.....	77.4	1.3	1.6	19.2	4.3	.5	450
	as purchased.....	.....	25.0	58.0	1.0	1.2	14.4	.....	.4	335
Huckleberries—	edible portion.....	1	.....	81.9	.6	.6	16.6	.....	.3	345
Lemons—	edible portion.....	4	.....	89.3	1.0	.7	8.5	1.1	.5	205
	as purchased.....	.....	30.0	62.5	.7	.5	5.9	.....	.4	145
Muskmelons—	edible portion.....	1	.....	89.5	.6	.....	9.3	2.1	.6	185
	as purchased.....	.....	50.0	44.8	.3	.....	4.6	.....	.3	90
Oranges—	edible portion.....	23	.....	86.9	.8	.2	11.6	.....	.5	240
	as purchased.....	.....	27.0	63.4	.6	.1	8.5	.....	.4	170
Pears—	edible portion.....	2	.....	84.4	.6	.5	14.1	2.7	.4	295
	as purchased.....	.....	10.0	76.0	.5	.4	12.7	.....	.4	260
Pineapple—	edible portion.....	1	.....	89.3	.4	.3	9.7	.4	.3	200
Plums—	edible portion.....	3	.....	78.4	1.0	.....	20.1	.....	.5	395
	as purchased.....	.....	5.0	74.5	.9	.....	19.1	.....	.5	370
Prunes—	edible portion.....	24	.....	79.6	.9	.....	18.9	.....	.6	370
	as purchased.....	.....	5.8	75.6	.7	.....	17.4	.....	.5	335
Raspberries—	as purchased.....	1	.....	85.8	1.0	.....	12.6	2.9	.6	255
Strawberries—	edible portion.....	22	.....	90.4	1.0	.6	7.4	1.4	.6	180
	as purchased.....	.....	5.0	85.9	.9	.6	7.0	.....	.6	175
Watermelon—	edible portion.....	2	.....	92.4	.4	.2	6.7	.....	.3	140
	as purchased.....	.....	59.4	37.5	.2	.1	2.7	.....	.1	60

The following analyses of apples made by Browne * are of interest. The first four analyses show the changes that occur in the composition of a Baldwin apple at different stages of its growth. Below these is given the average of the analysis of 160 samples, representing 27 varieties of apples.

COMPOSITION OF A BALDWIN APPLE AT DIFFERENT PERIODS.

Condition.	Water.	Solids.	Invert Sugar.	Sucrose.	Total Sugar.	Total Sugar after Inversion.	Starch.	Free Malic Acid.	Ash.	Sugar Coefficient.
Very green..	81.53	18.47	6.40	1.63	8.03	8.11	4.14	1.14	0.27	47.16
Green.....	79.81	20.19	6.46	4.05	10.51	10.72	3.67	....	....	53.10
Ripe.....	80.36	19.64	7.70	6.81	14.51	14.87	0.17	0.65	0.27	75.71
Over-ripe...	80.30	19.70	8.81	5.26	14.07	14.35	....	0.48	0.28	72.84

* Penn. Dept. of Agriculture, Bulletin 58.

AVERAGE COMPOSITION OF 27 VARIETIES OF APPLES.

Water	83.57
Solids	16.43
Invert sugar	7.92
Sucrose	3.99
Total sugar	11.91
Total sugar after inversion	12.12
Free malic acid	0.61
Ash	0.27
Sugar coefficient	73.76

The composition of the commoner nuts is shown in the following table:*

NUTS.

		Number of Analyses.	Refuse.	Water.	Protein.	Fat.	Total Carbohydrates.	Crude Fiber.	Ash.	Fuel Value per Pound Cal's.
Almonds—	edible portion.	11	.....	4.8	20.0	54.9	17.3	2.0	2.0	3030
	as purchased..	.....	45.0	2.7	11.5	30.2	9.5	.....	1.1	1660
Beechnuts—	edible portion.	1	.....	4.0	21.9	57.4	13.2	.....	3.5	3075
	as purchased..	.....	40.8	2.3	13.0	34.0	7.8	.....	2.1	1820
Brazil-nuts—	edible portion.	1	.....	5.3	17.0	66.8	7.0	.....	3.9	3265
	as purchased..	.....	49.6	2.6	8.6	33.7	3.5	.....	2.0	1655
Butternuts—	edible portion.	1	.....	4.4	27.9	61.2	3 5	.....	2.9	3165
	as purchased..	.....	86.4	.6	3.8	8.3	.5	.....	.4	430
Chestnuts, fresh—	edible portion.	9	.....	45.0	6.2	5.4	42.1	1.8	1.3	1125
	as purchased..	.....	16.0	37.8	5.2	4.5	35.4	.....	1.1	945
Cocoanuts—	edible portion.	1	.....	14.1	5.7	50.6	27.9	.....	1.7	2760
	as purchased..	.....	48.8	7.2	2.9	25.9	14.3	.....	.9	1413
Filberts—	edible portion.	1	.....	3.7	15.6	65.3	13.0	.....	2.4	3290
	as purchased..	.....	52.1	1.8	7.5	31.3	6.2	.....	1.1	1575

but little space need be given them beyond a résumé of their composition, and an outline of methods of proximate analysis applicable to their examination for food values. When, however, these products undergo the various processes incidental to their treatment for long keeping, such as preserving, canning, drying, pickling, and mixing with other ingredients, it is then that many varieties of fraudulent adulteration are practiced. Vegetable foods thus prepared form the subject of a separate chapter. Besides the proximate components that commonly occur in vegetable products, there are two other substances worthy of mention found in unripe vegetables and fruits, viz., inosite and pectose.

Inosite, $C_6H_{12}O_6,H_2O$, is not a carbohydrate, but, according to Hammersten, is an aromatic compound. Besides occurring in unripe fruits, it is found in green asparagus and beans.

Pectose is a substance the exact nature of which has not been fully determined, though it is thought to be a carbohydrate. It gives to unripe fruits and vegetables their peculiar hardness, and furnishes the basis for their gelatinous constituents. When the vegetable or fruit ripens, the insoluble pectose is transformed by the action of acids and possibly of ferments into *pectin*, a vegetable jelly, which gives to fruit juice the property of gelatinizing when boiled.

Inulin, $C_6H_{10}O_5$, is a starch-like substance, occurring in traces in chicory, and in the tubers of the potato and of the artichoke. It is a white, starch-like powder, slightly soluble in cold, and readily soluble in hot water, and converted into levulose by boiling with water, or by the action of acids.

METHODS OF PROXIMATE ANALYSIS.

Preparation of the Sample.—Cereals and dry leguminous foods are prepared for analysis by grinding in a coffee- or spice-mill to such a degree of fineness that the powder will pass through a sieve with 60 meshes to the inch. Green vegetables, beets, green peas, etc., are best reduced to suitable form for analysis by running through a domestic grinding-machine of the kind ordinarily employed in the kitchen for grinding and shredding meats and vegetables, being by this means reduced to a pulp of uniform consistency.

The following methods are based for the most part on those of the Association of Official Agricultural Chemists, employed for the analysis of foodstuffs with modifications.*

* U. S. Dept. of Agric., Div. of Chem., Bul. 46, revised.

Moisture.—From 2 to 3 grams of the finely divided substance are spread evenly over the bottom of a shallow platinum dish or watch-glass, and dried at 100° C. in an air-oven for five hours.

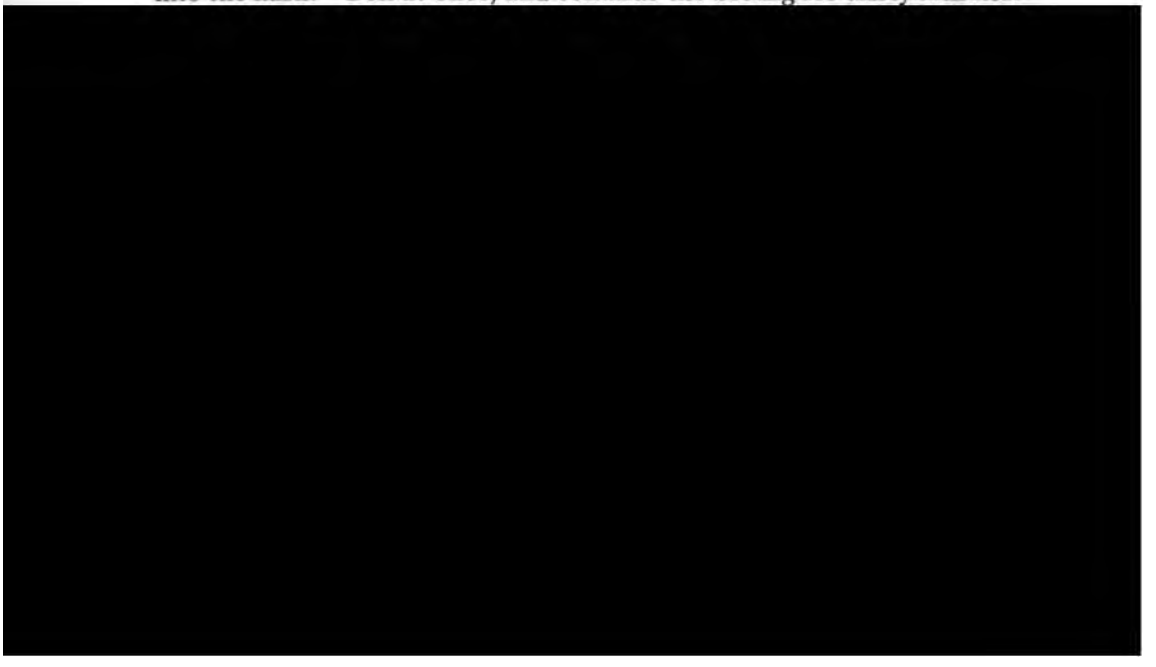
Ash.—The residue from the moisture determination (if a platinum dish has been used) is burned in the original dish to whiteness at the lowest possible red heat.* If a white ash cannot be obtained in this manner, exhaust the charred mass with water, collect the insoluble residue on a filter, burn, add this ash to the residue from the evaporation of the aqueous extract, and heat the whole to a low redness till the ash is white or nearly so.

Ether Extract.—2 to 3 grams of the sample are dried as for the determination of moisture, and extracted for sixteen hours with anhydrous, alcohol-free ether in a Soxhlet extractor. Dry the extract to constant weight, or the ether extract may be determined indirectly from the difference in weight of the dried substance before and after extraction, weighing it for convenience in the extraction tube.

Proteids.—The total nitrogen is determined according to the Gunning method in the absence of nitrates, using 0.5 gram of the finely divided substance. The total proteids are calculated by multiplying the total nitrogen by the appropriate factor, which varies with the different cereals. The factors for the common cereals are as follows: wheat, 5.70; rye, 5.62; oats, 6.31; corn, 6.39; and barley, 5.82.

Total Carbohydrates.—These are roughly determined by difference.

Crude Fiber (*Cellulose*)†.—The residue from the determination of the ether extract is transferred to a 500-cc. flask and 200 cc. of boiling 1.25% sulphuric acid are added. Connect the flask with an inverted condenser, the tube of which passes only a short distance beyond the rubber stopper into the flask. Boil at once, and continue the boiling for thirty minutes.



be made on a second piece of filter-paper to show the loss occasioned by treatment with alkali, and the necessary correction should be made.

The filter used for the first filtration may be linen, one of the forms of glass wool or asbestos filters, or any other form that secures clear and reasonably rapid filtration. A gooch was originally prescribed for the final filtration, but with many substances is apt to clog. The solutions of sulphuric acid and sodium hydroxide are to be made up of the specified strength determined accurately by titration, and not merely from specific gravity.

CARBOHYDRATES OF CEREALS AND VEGETABLES.

Classification.—As a rule the same carbohydrates are found in all cereals, being present, however, in varying proportions. By far the greater part of the carbohydrate content of cereals is starch, the other carbohydrates being comparatively small in amount, so that in rough work it is sometimes customary, though incorrect, to assume the entire amount of so-called "nitrogen-free extract" or carbohydrates (as determined by difference) to be starch.

The carbohydrates occurring in cereals may be classified as follows:

Principal carbohydrates of cereals:
- Insoluble.....
  - Starch
  - Cellulose
  - Pentosans
- Soluble.....
  - Sucrose
  - Dextrose
  - Dextrin
  - Raffinose (traces)

STARCH $(C_6H_{10}O_5)_n$.—Pure starch is a glistening, white, granular powder having a peculiar feeling when rubbed between the thumb and finger. It is a very hygroscopic, commercial starch containing about 18% of moisture. Starch is very widely distributed in the vegetable kingdom, occurring in almost every plant at some stage in its growth.

Starch is insoluble in cold water, alcohol, and ether; it is soluble in hot water, though not without change. By boiling with dilute acids, starch is first converted by hydrolysis into a mixture of dextrin and maltose, and finally by prolonged boiling into dextrose. Malt extract also hydrolizes starch in solution.

Detection.—Starch is best detected, when present to any appreciable extent in any mixture, by boiling a portion of the sample in water, cooling, and applying a solution of iodine. A characteristic blue color is produced if starch is present. Very small amounts of starch are best iden-

little groups or masses that at first sight might be mistaken for large grains; careful examination, however, shows the dividing lines.

Buckwheat Starch (Fig. 128, Pl. II, and Fig. 129, Pl. III).—This is a very common adulterant of many spices, especially pepper, which, as shown in Fig. 256, Pl. XXXIV, it much resembles in the manner in which its aggregate masses of granules group themselves. The individual granules are quite uniform in size, averaging about 0.006 mm. in diameter, and the masses are usually with difficulty broken up into individual grains, requiring considerable rubbing out under the cover-glass.

Rice Starch (Fig. 143, Pl. VI).—The granules of rice starch are considerably smaller than those of buckwheat, and are readily distinguished from the latter also by reason of the fact that they are much more sharply pointed (having less obtuse angles), and are grouped in smaller masses.

Starches of the Pea and Bean.—The starches of these legumes much resemble each other, and are with difficulty distinguished one from the other (see Fig. 164, Pl. XI, and Fig. 154, Pl. IX). The granules are more nearly oval than most other starches, and have both concentric rings and very marked hila. The starch of the pea is perhaps more regular and uniform in the size and form of its granules than that of the bean. Both peas and beans roasted are commonly used as adulterants of coffee.

Arrowroot.—There are many varieties of this starch, including Jamaica, Bermuda, East Indian, Australian, and others, all having certain variations in form and size, but resembling each other in a general way. Fig. 167, Pl. XII, shows the Bermuda arrowroot, the granules of which are somewhat egg-shaped, being usually smaller at one end than the other, and having rifted hila near the small end.



purity conveniently at hand, and in all doubtful cases these should be referred to for comparison.

Wheat Starch (Fig. 152, Pl. VIII).—This starch is very commonly used as an adulterant of mustard, ginger, cocoa, coffee, and other foods. Its granules are circular disks, occurring for the most part in two sizes, of which the larger vary from 0.021 mm. to 0.041 mm. in diameter, while the smaller average about 0.005 mm. The smaller granules are grouped irregularly in and around the larger, there being six to ten of the former to one of the latter. The larger granules are, however, the most distinctly characteristic, and are usually readily recognized in a mixture, not only by their shape, but by reason of the concentric rings with which they are provided, and which are generally but not always apparent.

Barley Starch (Fig. 124, Pl. I) —This much resembles wheat, in that it has two sizes of circular granules, but both sizes are respectively smaller than those of wheat, though present in about the same proportion. The larger granules vary from 0.013 mm. to 0.035 mm. in diameter, while the smaller average 0.003 mm. The concentric rings are less apparent in the barley than in the wheat.

Rye Starch (Fig. 148, Pl. VII) has also two sizes of circular disk-like granules, but the larger vary from 0.025 mm. to 0.05 mm. in diameter, and are considerably larger than the corresponding wheat granules. The smaller granules average about 0.004 mm. in diameter. There is also a much larger proportion of small granules present than in the case of wheat. The concentric rings are often very distinct in the large rye starch grains, and many of these show cross-shaped rifts in the center.

Corn Starch (Fig. 133, Pl. IV).—This starch is a common adulterant of spices, cocoa, and other foods. It is placed in a series of four cereal starches whose granules are polygonal, and all of which show more or less tendency to arrange themselves in close contact side by side in masses suggestive of a tessellated or mosaic floor. Arranged in order of the size of their grains, these starches are: Corn, oats, buckwheat, and rice. Corn starch granules tend toward the hexagonal in shape, varying from 0.007 mm. to 0.023 mm. in diameter, and having very marked rifted hila. They are most readily recognized in any mixture, and from their size are readily distinguishable from the other polygonal starches.

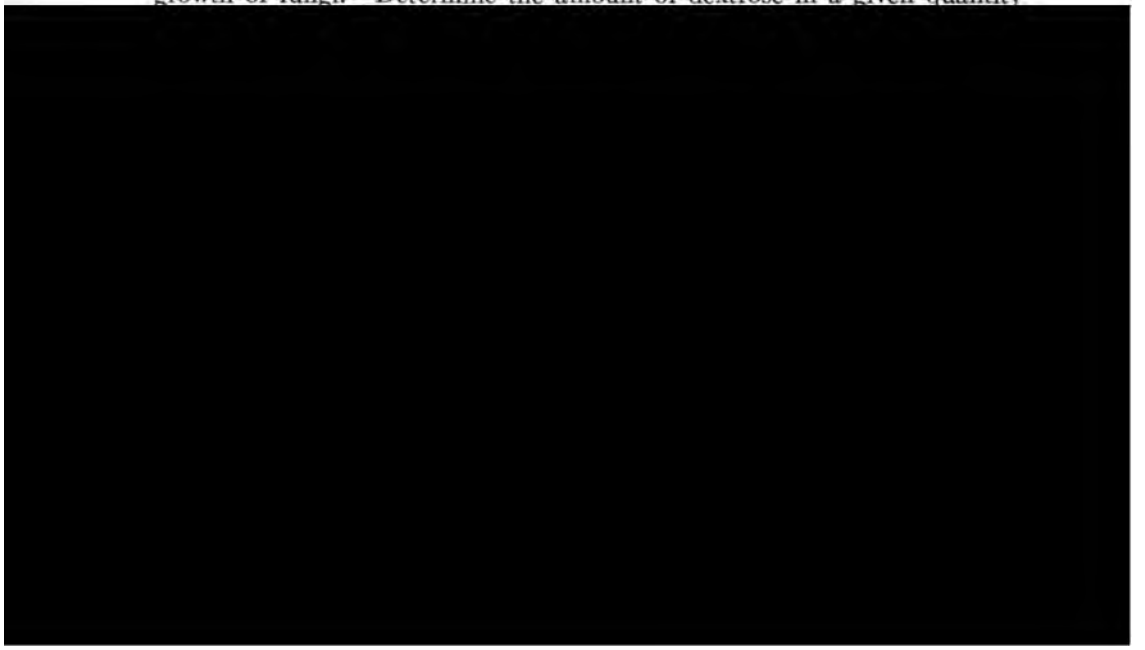
Oat Starch (Fig. 139, Pl. V).—The granules of this starch, averaging 0.004 mm. in diameter, are less regular in shape than the corn, besides being smaller. They have no rings or hila, and arrange themselves in

$$\underset{\text{Starch}}{12C_6H_{10}O_5} + 4H_2O = \underset{\text{Maltose}}{4C_{12}H_{22}O_{11}} + \underset{\text{Dextrin}}{2C_{12}H_{20}O_{10}}$$

$$\underset{\text{Maltose}}{C_{12}H_{22}O_{11}} + H_2O = \underset{\text{Dextrose}}{2C_6H_{12}O_6} \qquad \underset{\text{Dextrin}}{C_{12}H_{20}O_{10}} + 2H_2O = \underset{\text{Dextrose}}{2C_6H_{12}O_6}$$

Exhaust 3 grams of the finely divided substance with ether and alcohol as in the acid conversion method, wash the residue into a beaker with 100 cc. of water and boil directly for 30 minutes, stirring constantly and restoring the water lost on evaporation. Cool to 55° C., add 20 cc. of malt extract (prepared as below), and maintain at this temperature for one hour with occasional stirring. Boil a second time for 15 minutes, cool again to 55° C., and digest once more with 10 cc. of additional malt extract. The treatment with malt converts the starch into dextrin and maltose. Heat to boiling a third time, cool and make up to 250 cc. Filter, transfer 200 cc. of the filtrate to a 500 cc. flask, and add 20 cc. of hydrochloric acid (specific gravity 1.125), connect with a reflux condenser and heat in a boiling-water bath for two and one-half hours, by which process the dextrin and maltose are converted into dextrose. Cool, neutralize carefully with sodium hydroxide (avoiding an excess), clarify if necessary with 10 to 20 cc. of alumina cream (p. 482), and make up to 500 cc. Mix well, pour through a dry filter, and determine the dextrose in an aliquot part of the filtrate, using the factor 0.9 for converting dextrose to starch. Correct for the copper reducing power of the malt extract, as below.

Preparation of Malt Extract.—Dry malted barley can be readily obtained from any brewery. Treat 15 to 20 grams of freshly pulverized malt for several hours with 100 cc. of water, shaking occasionally. Filter the solution, and add two or three drops of chloroform to prevent the growth of fungi. Determine the amount of dextrose in a given quantity



Cellulose turns violet when treated with chloriodide of zinc, and blue when treated with sulphuric acid and iodine in potassium iodide (p. 79).

The "crude fiber" as determined in foods, being the portion that resists the action of hot dilute acid and alkali, is composed largely of cellulose.

The Pentosans are of comparatively small importance, and have been little studied. They are amorphous in character, insoluble in water, but soluble in dilute alkali, and are capable of conversion by boiling with dilute acids into so-called pentose sugars, the best known of which are xylose and arabinose, corresponding to the pentoses xylan and araban respectively. Strictly speaking the term "hemicellulose" is the more appropriate generic term to apply to the insoluble carbohydrate bodies which are capable of hydrolysis by acids to sugars, inasmuch as there are insoluble bodies besides the pentosans that may thus be converted into sugar, such as the hexosans, hydrolyzed by acid to hexose sugars, mannose, galactose, etc. The term "wood gum" is also used synonymously with hemicellulose. Since the greater portion of these insoluble hydrolizable carbohydrates are pentosans, it is simpler to calculate them all as such.

Determination of Pentosans.—Pentosans are determined either by hydrolyzing to reducing sugar, and estimating the latter as described on page 228 (Stone's method) or by calculation from the furfurol * yielded by them on distillation of the sample with hydrochloric acid, as carried out in the provisional method of the A. O. A. C.† as follows:

Three grams of the material are placed in a flask, together with 100 cc. of 12% hydrochloric acid (specific gravity 1.06) and several pieces of recently heated pumice stone. The flask, placed upon wire gauze, is connected with a condenser, and heat applied, rather gently at first, using a gauze top to distribute the flame, and so regulated as to distill over 30 cc. in about ten minutes. The 30 cc. driven over are replaced by a like quantity of the dilute acid, and the process continued so long as the distillate gives a red color when applied to a filter-paper soaked in anilin acetate. To the completed distillate is gradually added a quantity of phloroglucin (free from diresorcin) dissolved in 12% hydrochloric acid, and the re-

* Furfurol or furfuraldehyde ($C_5H_4O_2$) is the aldehyde of pyromucic acid. It is a colorless liquid, having an odor suggestive of cassia. Its boiling-point is 162° and its specific gravity 1.164. It is sparingly soluble in water and readily soluble in alcohol. Nearly half the tissue of ordinary bran, exclusive of proteids and starch, yields furfurol on distillation with acid.

† U. S. Dept. of Agric., Div. of Chem., Bul. 46, p. 25.

sulting mixture is thoroughly stirred. The amount of phloroglucin used should be about double that of the furfurol expected. The solution first turns yellow, then green; and very soon an amorphous greenish precipitate appears, which grows rapidly darker, till it finally becomes almost black. The solution is made up to 500 cc. with 12% hydrochloric acid and allowed to stand over night.

The amorphous, black precipitate, a condensation product the exact composition of which is unknown, is filtered into a tared gooch through an asbestos felt, washed with 100 cc. of water, dried to constant weight by heating from three to four hours at 100°, cooled and weighed, the increase in weight being reckoned as phloroglucide. To calculate the furfurol from the phloroglucide, use the following formulæ:

Phloroglucide (less than and up to 0.2 gram) ÷ 1.82 = furfurol.
Phloroglucide (from 0.2 to 0.3 gram) ÷ 1.895 = furfurol.
Phloroglucide (from 0.3 to 0.4 gram) ÷ 1.92 = furfurol.
Phloroglucide (above 0.4 gram) ÷ 1.93 = furfurol.

To calculate the furfurol to pentosan or pentose use the following formulæ:

I. (furfurol 0.0104) × 1.68 = xylan.
II. (furfurol 0.0104) × 2.07 = araban.
III. (furfurol 0.0104) × 1.88 = pentosan.
IV. (furfurol 0.0104) × 1.91 = xylose.
V. (furfurol 0.0104) × 2.35 = arabinose.
VI. (furfurol 0.0104) × 2.13 = pentose.

The reactions that take place are thought to be somewhat as follows:



and add a few drops of concentrated sulphuric acid. If more than a faint violet color is produced, the phloroglucin is unfit for use.

SEPARATION AND DETERMINATION OF THE VARIOUS CARBOHYDRATES OF CEREALS, ETC. STONE'S METHOD.

Stone has thus tabulated the results of a series of analyses of various samples of wheat, flour, corn, and bread, in which he has separated the principal carbohydrates.* Later methods give higher starch figures.

PERCENTAGES OF VARIOUS CARBOHYDRATES IN CERTAIN FOODSTUFFS.

	Sucrose.	Invert Sugar	Dextrin.	Soluble Starch.	Normal Starch.	Pento-sans.	Crude Fiber.
Whole wheat, I	0.52	0.08	0.27	0.00	30.94	4.54	2.68
Whole wheat, II	0.72	0.00	0.41	0.00	30.36	4.37	2.51
Wheat flour, I	0.18	0.00	0.90	0.00	46.19	0.00	0.25
Wheat flour, II	0.20	0.00	1.06	0.00	34.04	0.00	0.25
Corn	9.27	0.00	0.32	0.00	42.50	5.14	1.99
Sugar-beet	8.38	0.07	0.35	0.00	0.00	4.89	1.00
Bread (wheat, I)	0.05	0.32	0.68	1.37	27.93	4.16	2.70
Bread (wheat, II)	0.06	0.37	0.23	2.36	27.08	4.34	2.02
Bread (flour, I)	0.01	0.10	0.27	1.99	34.70	0.00	0.34
Bread (flour, II)	0.15	0.38	0.91	1.74	31.99	0.00	0.17
Corn cake (maize)	0.16	0.19	0.00	2.80	40.37	3.54	2.22

Determination of Cane Sugar.—100 grams of the finely ground material are extracted by boiling under a reflux condenser with 500 cc. of 95% alcohol for three hours, the alcoholic extract is filtered, evaporated nearly to dryness, and then taken up with a small amount of water, to separate the sugar from the oils and waxes dissolved by the alcohol. This aqueous solution is invariably dextro-rotary, and seldom contains any reducing sugar. If the latter is present, it is determined in an aliquot part of the aqueous solution with Fehling's solution, the result being calculated to dextrose. The remainder of the aqueous sugar solution, or the whole of it, if, as is almost always the case, dextrose is absent, is then inverted by heating with hydrochloric acid in the usual manner (page 483) and the sugar is estimated with Fehling's solution, calculating the result to sucrose (page 497).

Determination of Dextrin.—Digest the residue from the above alcoholic extraction from eighteen to twenty-four hours with 500 cc. of cold distilled water, shaking frequently. On filtering, a clear solution is ob-

* Jour. Am. Chem. Soc., XIX, 1897, p. 183, and U. S. Dept. of Agric., Off. of Exp. Sta., Bul. 34.

tained, which should be tested with iodine for soluble starch. If the latter is not found (which is nearly always the case), the solution is concentrated to a small volume, avoiding a temperature higher than 80° to 90°, and this is boiled under a reflux condenser for two hours with one-tenth its volume of hydrochloric acid (specific gravity 1.125). Determine the dextrose by Fehling's solution and calculate to dextrin by the factor 0.9. Or, instead of submitting the concentrated aqueous extract to hydrolysis as above, the dextrin may be roughly determined gravimetrically therein by treating with several volumes of strong alcohol until no further precipitation is produced. The flocculent precipitate thus obtained is collected, dried, and weighed.

Determination of Starch.—Dry in an oven the residue from the preceding treatment and determine its quantitative relation to the original sample; 2 grams are then accurately weighed and subjected to the diastase method of starch determination (page 223).

Determination of Pentosans and Hemicelluloses.—The washed residue, left after filtering off the starch-containing solution from the process of heating with malt extract in the preceding starch determination, is boiled for an hour with 100 cc. of 1% hydrochloric acid, which converts all the pentosans into sugar. Filter, and wash the residue thoroughly, make up the solution to 200 cc., and determine the sugar with Fehling's solution, calculating the results for xylan, assuming that the chief sugar formed is xylose. The reducing power of xylose is assumed to be 4.61 milligrams for each cubic centimeter of Fehling's solution. If the volumetric Fehling method is used, 10 cc. of Fehling's solution are thus equivalent to 0.046 gram xylose. Xylose × 0.88 = xylan.

Crude Fiber (*Cellulose*).—The residue from the last dilute acid



for separation of the vegetable proteids. For fuller details the reader is referred to the work of Osborne *et al.* in the American Chemical Journal, Vols. 13, 14, and 15, and to the Journal of the American Chemical Society, Vols. 17, 18, 19, and 20.

Proteids Soluble in Water and Dilute Salt Solution.—By the action of various solvents it is possible to separate the different classes of proteids for examination or analysis. Thus water at first applied extracts certain of the soluble proteids, as does a weak salt solution. Osborne and Voorhees recommend the use of a 10% solution of sodium chloride as the first solvent to apply for separating vegetable proteids, shaking the finely ground material with twice its weight of the salt solution. The salt solution, after filtering, is then subjected to dialysis, the proteid matter thus separated out being a globulin, while that not precipitated on dialysis is assumed as the proteid matter of the substance soluble in water. Two albumins and a proteose are found in wheat to be thus soluble in water.

If the proteids soluble in salt solution are to have their total nitrogen determined, they are completely precipitated from the solution by saturating with zinc or ammonium sulphate.

There are thus two classes of proteids soluble in 10% salt solution: (*a*) globulins, insoluble in water alone, and (*b*) albumins and proteoses, which are soluble in water.

Separation of Albumins, Proteoses, and Globulins.—Starting with the aqueous solution containing the albumins and proteoses, if present, the former are best separated according to Osborne and Vorhees by fractional coagulation, effected by heating at different temperatures, those that precipitate out at a temperature under 65° being first filtered out, and the filtrate submitted to a higher temperature not exceeding 85°. The two portions thus separated may be collected in filters, and their nitrogen separately determined.

The proteose may be precipitated from the filtrate by saturating with ground salt, or by adding, first salt to the extent of 20%, and finally acetic acid.

The globulins, precipitated in the original 10% salt solution by the process of dialysis as described, may themselves be separated by employing salt solution of varying strength as solvents.*

Proteids Soluble in Dilute Alcohol, but Insoluble in Water.—The residue from the treatment with 10% sodium chloride is digested with 75% alcohol at about 46° C. for some time and filtered. The residue is further

* Am. Chem. Jour., 13, p. 464.

digested at about 60° with 75% alcohol three separate times. The evaporated filtrates contain the alcohol-soluble proteids. In this class are the hordein of barley, the gliadin of wheat and rye, and the zein of corn.

Proteids Insoluble in Water, Salt Solution, and Dilute Alcohol.—It is customary to determine the nitrogen in the final residue without further attempt to separate the remaining proteid matter. It is, however, possible to further extract with alkaline and acid solvents, if desired, which process, however, changes the nature of the proteids from that in which they originally exist in the substance.

Character and Amount of Proteids in Wheat.*—The proteids of wheat, according to Osborne, are five in number as follows:

		Amount Present, Per Cent.
Soluble in water.	Albumin (leucosin)	0.3 to 0.4
	Proteose	0.3
Soluble in 10 per cent NaCl:	Globulin (edestin)	0.6 to 0.7
Soluble in dilute alcohol:	Gliadin	4.25
Insoluble in above:	Glutenin	4.00 to 4.5

Gluten.—This term is applied to the entire proteid content of wheat flour insoluble in water, and the value of flour for baking bread depends on the amount of gluten present. Gluten contains the two definite proteids *gliadin* and *glutenin.* Crude gluten is a complex mixture of many bodies, containing, besides the two proteids above named, small quantities of cellulose, mineral matter, lecithin, and starch.

Wiley † recommends the following method for separating crude gluten from flour. Ten grams of the fine-ground flour are placed in a porcelain dish, well wet with nearly an equal weight of water at a temperature not to exceed 15°, and the mass worked into a ball with a spatula, taking care that none of it adheres to the walls of the dish. The ball of dough



Separation and Determination of Wheat Proteids.—*Teller's Method.**—***Non-gluten Nitrogen.***—Two grams of the finely divided sample are mixed with about 15 cc. of 1% salt solution in a 250-cc. flask. The flask is shaken at intervals of ten minutes during one hour, after which it is filled to the mark with the salt solution and allowed to stand two hours. The supernatant liquid is then filtered through a dry filter into a dry flask, leaving most of the solid material in the flask, passing the first part through twice, if necessary, for a clear filtrate. With a pipette, exactly 50 cc. of clear filtrate are run into a 500-cc. Kjeldahl digestion-flask, 20 cc. of the usual reagent sulphuric acid for the Gunning process (p. 61) are added, and the contents of the flask brought to a gentle boil. After the water has been driven off and the acid has stopped foaming, the potassium sulphate is added and the digestion completed. From the per cent of nitrogen thus obtained 0.27% is deducted, this figure corresponding to the amount of gliadin soluble in 1% salt solution under the above conditions. The remainder is the percentage of non-gluten nitrogen.

Gluten Nitrogen.—This is obtained by difference between the total nitrogen and the non-gluten nitrogen as above obtained, or by deducting the combined nitrogen of the edestin, leucosin, and the amido-nitrogen from the total nitrogen.

Edestin and Leucosin.—Edestin is a globulin belonging to the vegetable vitellins, and is precipitated from salt solutions by dilution, or by saturation with magnesium or ammonium sulphate, but not by saturating with sodium chloride. It is not coagulated below 100° C., but is partly precipitated by boiling. Leucosin is an albumin, coagulating at 52°, but precipitates from salt solution by saturating with sodium chloride or magnesium sulphate.

To 50 cc. of the clear salt extract, obtained as described under non-gluten nitrogen, 250 cc. of pure 94% alcohol are added in a Kjeldahl 500-cc. digestion-flask, the contents thoroughly mixed, and allowed to stand over night. The precipitate is collected in a 10-cm. filter, which is returned to the flask and the nitrogen determined. This represents the nitrogen of the combined edestin and leucosin. These proteids may, however, be separated by coagulating the leucosin at 60°, and precipitating the edestin by adding alcohol to 50 cc. of the clear filtrate, determining the nitrogen separately in each precipitate.

Amido-nitrogen.—Allantoin, asparagin, cholin, and betaine are nitrogenous bases present in wheat.

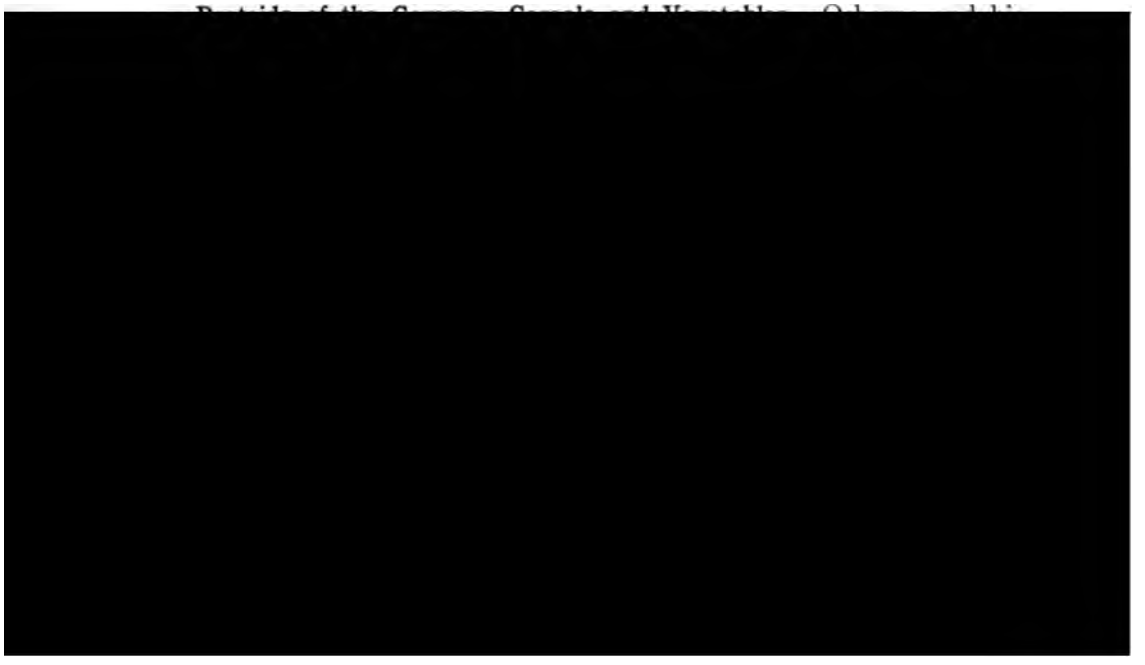
* Ark. Exp. Sta. Bul. 42, p. 96.

Ten cc. of a 10% solution of pure phosphotungstic acid are added to 100 cc. of the clear salt extract as above obtained, thus precipitating all the proteids, which are allowed to settle preferably over night. Filter, and determine the nitrogen in the clear filtrate. The filtrate should be tested with a little of the phosphotungstic acid reagent to make sure that all the proteids have been separated. In some cases, as in bran for instance, more than 10 cc. of the reagent are necessary.

Gliadin is dissolved most readily from flour by hot dilute alcohol, but is entirely insoluble in absolute alcohol. One gram of the material is extracted with 100 cc. of hot 75% alcohol, by shaking the mixture thoroughly in a flask, and heating for an hour at a temperature just below the boiling-point of alcohol, with occasional shaking. After standing for an hour, the hot liquid is decanted upon a 10-cm. filter, and 25 cc. of the hot alcohol are added to the residue and shaken, after which the residue is again allowed to settle, and the liquid decanted. This is repeated six times. The remainder of the alcohol is then driven off by evaporation, and the nitrogen determined in the residue. The difference between the total nitrogen and the nitrogen thus obtained, gives the nitrogen of the alcoholic extract, which includes the amides. Subtracting the latter, or amido-nitrogen, the remainder is the gliadin nitrogen.

Glutenin Nitrogen.—This is the difference between the gluten nitrogen and the gliadin nitrogen.

The factor by which the nitrogen should be multiplied in determining the various proteids, according to Osborne and Voorhees, is 5.7 for wheat.



	Per Cent.
Proteids of barley:*	
Soluble in water: { Leucosin / Proteose }	0.3
Soluble in salt solution, edestin	1.95
Soluble in dilute alcohol, hordein	4.00
Insoluble in water, salt solution, and alcohol	4.50
Proteids of corn:†	
Soluble in water: Proteose	0.06
Soluble in salt solution: Very soluble globulin	0.04
Soluble in salt solution: Maysin	0.25
Soluble in salt solution: Edestin	1.10
Soluble in dilute alcohol: Zein	5.00
Insoluble in above, but soluble in two-tenths per cent potash solution	3.15
Proteids of pea:‡	
Soluble in salt solution: Globulins { Legumin	7.00
Soluble in salt solution: Globulins { Vicilin	3.00
Soluble in water: Albumin, legumelin, proteose	2.03

Proteids of Potato.§—Almost the whole proteid content of the potato consists of a globulin to which Osborne has applied the name "tuberin." Proteose is also present in very small amount.

Composition of the Ash of Cereals and Vegetables.—A complete analysis showing the definite composition of the ash of cereals and vegetable products is not often necessary. When, however, this is to be done, it is of importance to obtain as large an amount of the ash as possible for examination, and to this end the dried, finely divided material is burnt in a muffle at a low red heat in platinum dishes‖ of at least 50 grams capacity, adding from time to time 25 grams of the material, whenever the bulk of the burnt residue is sufficiently reduced to admit of it. This is continued until enough ash is obtained for the work in hand, and the heat is applied till the product is light gray in color.

The detection and estimation of the various common ash ingredients are carried out by the qualitative and quantitative processes commonly employed in mineral analysis, and will not be given here.

The following table¶ shows the composition of the pure ash of common cereals:

---

* Jour. Am. Chem. Soc., 17, p. 539.

† *Ibid.*, 19, p. 525.

‡ *Ibid.*, 18, p. 583; 20, pp. 348 and 410.

§ *Ibid.*, 18, p. 575.

‖ On account of the high content of phosphoric acid in cereals it is unsafe to ignite in platinum over a free flame. It may safely be done with caution at a moderate heat in a muffle. See p. 56.

¶ U. S. Dept. of Agric., Bur. of Chem., Bul. 13, part 9, p. 1212.

COMPOSITION OF ASH OF CEREALS.

	K_2O.	Na_2O.	CaO.	MgO.	Fe_2O_3.	P_2O_5.	SO_3.	Cl.	SiO_2.
Wheat (Canada)	24.03	9.55	3.50	13.24	0.52	46.87	0.01	0.00	2.28
Rye (Minnesota)	27.60	4.64	5.56	11.73	5.23	41.81	0.52	0.58	2.45
Barley (U. S.)	24.15	6.42	2.44	8.23	0.33	35.47	0.22	0.56	22.30
Oats (U. S.)	15.91	4.38	4.09	7.18	0.20	24.34	0.48	1.02	42.64
Corn (U. S.)	33.92	7.72	3.18	17.99	0.50	35.25	0.44	0.00	1.00
Rice, polished (Guatemala)	20.84	13.98	4.48	9.60	0.89	43.21	0.24	0.80	6.14
Buckwheat (U. S.)	35.15	2.26	6.62	20.55	1.68	24.09	3.59	0.67	5.54

Teller * obtained the following results of ash analyses of flour, bran, and wheat:

ASH OF WHEAT PRODUCTS.

	Patent Flour.	Straight Flour.	Low Grade.	Bran.	Wheat.
Silica	2.33	1.28	.50	.97	1.04
Alumina	.41	.15	.12	.07	.11
Ferric oxide	.47	.26	.25	.27	.27
Potash	38.50	36.31	32.27	28.19	29.70
Soda	0.00	0.00	0.00	0.00	0.00
Lime	5.59	5.65	4.51	2.50	3.10
Magnesia	4.39	6.44	9.33	14.76	13.23
Phosphoric acid	48.05	49.32	53.10	52.18	52.14
Sulphur trioxide	.16	.52	.00	.10	.22
Chlorine	.....	.....	.....	.01	.01
Zinc oxide	.....	.04	.....	.27	.24
Sum	99.90	99.97	100.08	99.95	100.06
Per cent of total ash	.31	.40	.70	.....	1.62

König gives the following analyses of the ash of various leguminous



FLOUR.

Flour is the term applied to the finely ground and bolted substance of wheat and other grains, though, unless otherwise qualified, by the term "flour" is generally understood that of wheat. In the process of manufacture, the dried wheat or grain is first crushed between millstones, forming the comparatively coarse product known as whole meal. This, by bolting, may be separated simply into flour and bran, but in the crude milling of years ago at least three products were usually obtained from wheat, viz., fine flour, coarser shorts, or middlings, and bran.

In the improved modern processes of milling, which meet the demands for the very finest flour, as well as other grades, the material is subjected to repeated sifting and grinding between grooved rollers, with the result that it is possible to turn out as many as ten separate grades of flour, as is shown by the following record of a mill near Trieste:

Groats,* A and B	2	per cent	
Flour, No. 0	5	"	
" " 1	12	"	
" " 2	6	"	41 per cent extra flour
" " 3	6	"	
" " 4	5	"	
" " 5	5	"	
" " 6	14	"	
" " 7	9	"	
" " 8	5	"	38 per cent medium and common
" " 9	10	"	
Bran	18	"	
Loss	3	"	21 per cent waste
	100	per cent	

Analyses of these separated products made by C. A. Pillsbury are as follows:

	Water.	Ash.	Phosphoric Acid.	Nitrogen.	Proteids Calculated.
Groats*	10.57	0.42	0.20	2.24	14.65
No. 0	10.37	0.43	0.14	1.68	10.76
" 1	10.23	0.41	0.21	1.68	10.76
" 2	10.47	1.03	0.22	1.72	11.02
" 3	10.07	1.02	0.17	1.72	11.02
" 4	[illegible]	1.19	0.25	1.74	11.15
" 5	9.66	0.69	0.35	1.80	11.54
" 6	[illegible]	1.04	0.24	1.84	11.79
" 7	10.99	0.81	0.21	1.80	11.54
" 8	9.86	1.01	0.36	1.90	12.18
" 9, coarse bran	9.71	7.32	2.14	1.98	12.69
" 10, fine bran	11.01	4.21	0.70	2.20	14.16

* Masses of the interior of the berry.

In this country the common practice of most mills is to produce about three grades of flour, somewhat as follows:

100 lbs. wheat..... {
- Patent or middlings flour, 55 lbs.
- Bakers' or family " 15 "
- Low-grade or bran " 6 "
- Bran, shorts, and waste (used principally for cattle food), 24 "

Graham Flour, or whole-wheat flour, is made from the unbolted meal of wheat, ground as finely as possible. It is actually a mixture of flour and bran.

Composition of Common Flours.—The following analyses are collated and summarized from Bulletin 13, part 9 of the Bureau of Chemistry:

	No. of Analyses.	Moisture.	Proteids, N×6.25.	Proteids, N×5.70.	Moist Gluten.	Dry Gluten.
Patent wheat flour.....	40	12.77	10.55	9.62	25.97	9.99
Common market wheat flour.....	19	12.28	10.18	9.28	24.55	9.21
Bakers' and family flour.....	14	11.69	12.28	11.20	34.70	13.07
Indian-corn flour.....	3	12.57	7.13			
Rye flour.....	1	11.41	13.56			
Barley flour	1	10.92	7.50			
Buckwheat flour	1	11.89	8.75			

	Ether Extract.	Ash.	Carbohydrates, N×6.25.	Crude Fiber.	Calculated Calories of Combustion.

branding them officially with the date as well as the grade. Marked quotations also are based on the standard terms adopted. The names of the various grades differ with the locality. In St. Louis the following names are adopted in order of their quality, viz., Patent, Extra Fancy, Fancy, Choice, and Family.

Such systems of inspection are under the auspices of local dealers, being organized and maintained for their own protection, and have not as yet been the subject of state or even municipal supervision, as in the case of meat inspection. The grade or quality of flour is determined largely by its appearance and color, by its fineness as indicated by rubbing between the fingers, by its odor, and by the so-called "doughing test," which consists in kneading the flour with water under fixed conditions, and noting its tenacity and elasticity.

Adulteration of Flour.—Besides the substitution of cheaper or inferior grades for those of higher quality, the fraudulent admixture of cereals other than wheat is not uncommon in flour. Corn meal is sometimes found as an adulterant of wheat flour. Its presence is best detected by the microscope, the difference between the wheat and corn starch being readily apparent.

Finely ground mineral adulterants are said to have been used in flours, but no authentic instance of this kind has come to the writer's knowledge. Any considerable admixture of such a nature would be manifest in the increased ash.

Alum in Flour.—The addition of alum to flour was formerly a common practice in Europe, both by miller and baker, for the purpose of improving the appearance of inferior or slightly damaged flour. Hence it was frequently found in the cheaper grades of flour and bread. Now, however, it is rarely if ever used for this purpose, and the presence of notable quantities of alum or its compounds in flour or bread is usually due to its use as an ingredient of leavening powders.

Detection.—To detect alum in flour, mix about 10 grams of the sample in 10 cc. of water and add about 1 cc. of an alcoholic tincture of logwood (5 grams logwood digested in 100 cc. alcohol) and about 1 cc. of a saturated solution of ammonium carbonate. Stir the whole well together. If the sample is pure, the color will be a faint brown or pink, but if alum is present, a distinct lavender-blue color is produced, which should remain after heating for two hours in the water-oven.

Alum may also be tested by the ammonium chloride method, described on page 263.

The Cold-water Extract of Flour.—The amount of cold-water extract shows to some extent whether or not a flour has been damaged, because in cases of damaged flour the starch grains have usually suffered injury, rendering them to some extent soluble. Wanklyn specified the following method of determining the cold-water extract.

One hundred grams of the flour are thoroughly mixed with distilled water in a graduated liter-flask, which is finally filled with water to the mark; the contents are thoroughly mingled by frequent shaking during six or eight hours, and allowed to stand over night. The supernatant liquid is then poured upon a filter. After rejecting the first few cubic centimeters of the filtrate, exactly 50 cc. are collected and evaporated to dryness in a tared platinum dish on the water-bath. The weight of the dried residue, multiplied by 20, gives the quantity of cold-water extract, which in a sound flour, according to Wanklyn, should not exceed 5%.

Gluten Flour.—This preparation is primarily intended for the use of diabetics, from whose dietary carbohydrates must be excluded; hence its value for their purpose is determined by its low content in starch.

The following analyses of commercial gluten preparations were made by Woods and Merrill.*

	Protein.	Fat.	Carbohydrates.	Ash.
"Cooked gluten"	16.88	3.86	76.80	2.46
Whole-wheat gluten	17.89	5.20	73.85	3.06
"Glutine"	15.31	0.99	82.52	1.17
Breakfast cereal gluten	43.70	1.60	44.40	0.70
Plain gluten flour	53.60	1.20	34.50	0.60
Self-raising flour	31.50	1.40	53.20	3.80



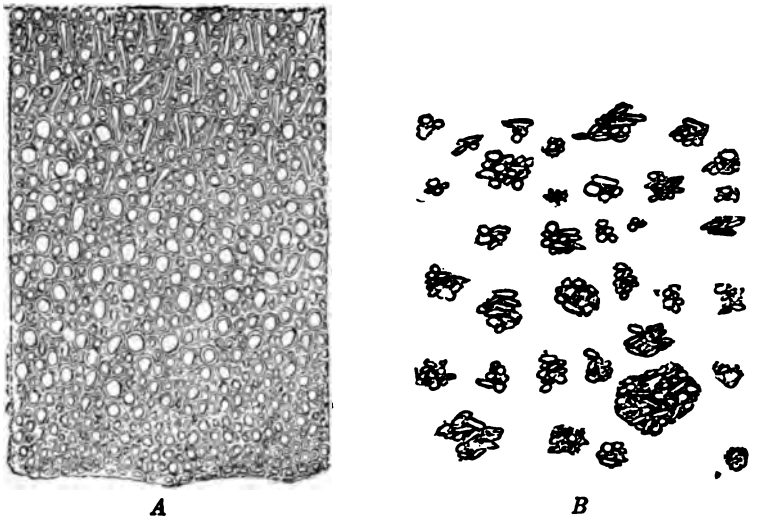
Moisture	10.78
Ash	2.20
Fat	3.25
Proteids	14.25
Sugars	1.70
Dextrin	2.55
Starch	56.55
Undetermined	8.72
	100.00

Ergot.—Ergot is a fungus growth that occasionally develops within the grain of rye and other cereals, and, unless care is taken, becomes ground with them in the preparation of meal and flour. Ergot contains alkaloids of a poisonous nature, and instances are on record of its accidental presence in cereal preparations causing serious injury to health. While most commonly found in rye, ergot occasionally grows in wheat. If flour or meal containing ergot be treated with a very dilute solution of anilin violet, the stain will be practically absorbed by the damaged particles of the grain, and resisted by the normal granules. If shaken with dilute alkali, a meal or flour contaminated with ergot is colored violet, which by treatment with acid turns red. A hot, alcoholic extract of flour containing ergot is colored red when treated with dilute sulphuric acid.

Ergot in ground cereal preparations is best recognized under the microscope, appearing as a fine network of mostly colorless parenchyma cells, containing globules of fat (Fig. 56). Some of the cells are circular, others considerably elongated, and some contain a deep-brown coloring matter, which, when treated with ammonia, takes a violet-red color. Occasionally the cell walls appear of a dark color. If the suspected sample be treated with dilute anilin violet, as above described, the stained ergot fragments will be especially apparent under the microscope.

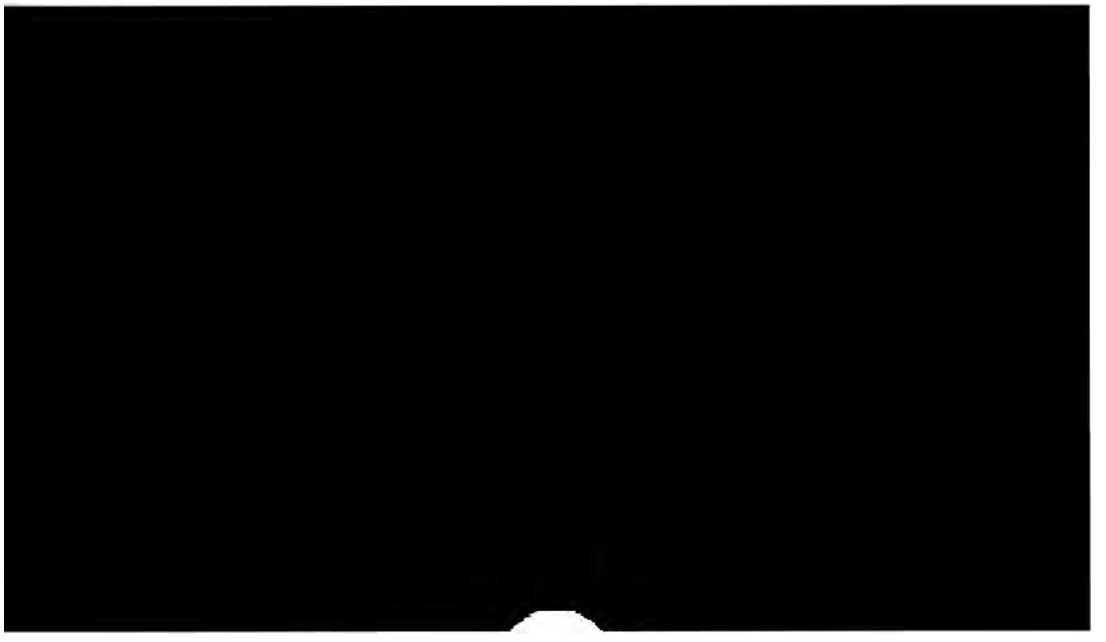
Microscopical Examination of Flour and Meal.—A study of the histology of the various cereal grains is beyond the scope of the present work, and the reader who wishes to pursue this branch of the subject is referred to the treatises of Vogl and Tsirsch and Osterle. The characteristics of the tissues of the various ground cereal grains are quite distinctive when carefully studied, sufficiently so, at least, to serve to identify a particular grain, when its flour is submitted in its purity to examination.

In a mixture of two or more flours, however the corresponding tissues of the different grains resemble each other so closely that it is by no means an easy task to distinguish them by the appearance of these tissues. In such a mixture the microscopical appearance of the starch furnishes the chief reliance on which to base a judgment as to the character of the grains. We have already seen that the various cereal starches differ considerably in morphology and mode of grouping from each other, and this is true to such an extent that the expert can readily identify them. Since starch furnishes much more than half the content of all ground



A *B*

FIG. 56.—*A*, Transverse Section of the Ergot of Wheat under the Microscope; *B* Powdered Wheat Ergot. (After Villiers and Collin.)



Wheat Flour or Meal.—Fig. 57 shows the elements typical of wheat meal.

e are the long six-sided cells of the epicarp, with bead-like walls; next to these are the elongated cells, *ct*, with similar bead-like walls, these cells usually running transversely to those of the epicarp; *p* are the single-celled hairs attached normally to a portion of the epicarp.

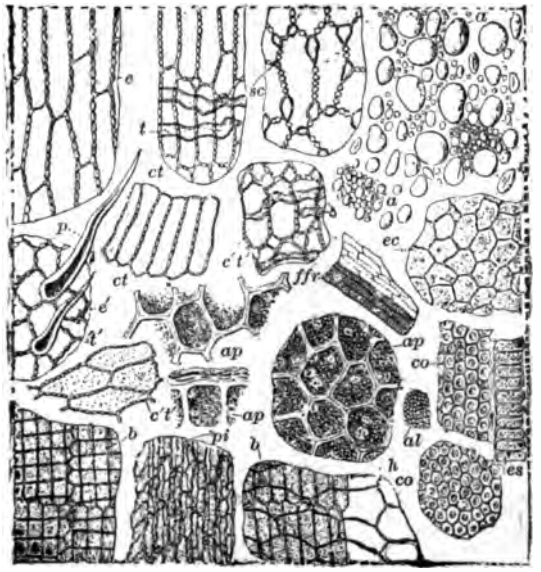


Fig. 57.—**Wheat Flour under the Microscope.** *e*, epicarp; *p*, hairs; *ct*, *c't'*, transverse cells; *t*, tubular cells; *b*, brown seed coat; *pi*, masses of pigment; *ec*, sheath around the embryo; *es*, outer envelope of the sheath; *co*, cotyledon; *ffv*, fibro-vascular ducts; *sc*, thickened cells; *a*, starch; *al*, aleurone; *ap*, proteid layer. (After Villiers and Collin.)

These hairs are somewhat distinctive, being conical or slightly tapering, often bent and enlarging into a bulb at the base. The thickness of the wall of the wheat hair is usually equal to the thickness of the lumen; *sc* are hardened or lignified cells of the mesocarp, often with intercellular spaces. The tubular cells, *t*, of the endocarp are rarely isolated, but more often attached to the transverse cells and running perpendicular thereto. *b* is the seed coat, of a yellowish or orange-brown color, having two layers, composed of very thin-walled cells, crossing each other at right angles. Fragments of the hyaline layer *h* are often found united with bits of the seed skin. A piece of the sheath or layer immediately surrounding the embryo or germ

is shown at *ec*, *es* being a palisade-like layer lying outside the sheath. The aleurone cells, sometimes incorrectly termed gluten cells, are represented at *ap*. These are polygonal, sometimes nearly square in plan view, and normally contain the aleurone grains *al*, but often are broken down, and, when seen in the flour, empty. The rounded, angular aleurone grains appear sometimes isolated and sometimes in masses. If there is danger of mistaking them for small starch grains, cochineal should

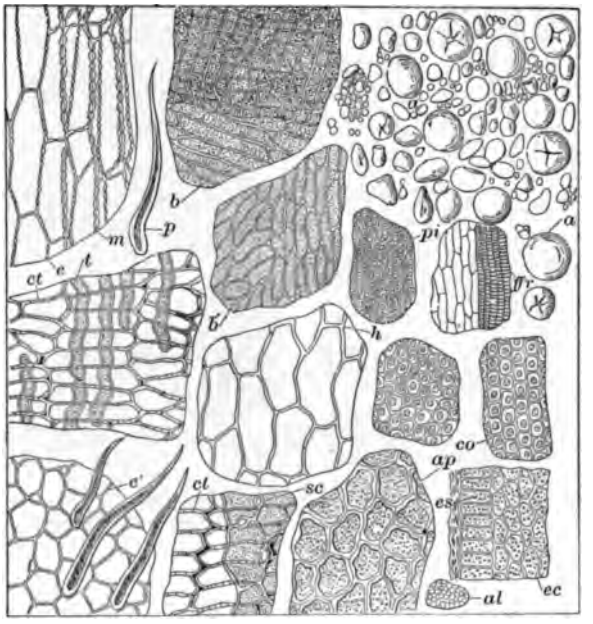


FIG. 58.—Rye Meal under the Microscope. *ee'*, epicarp; *m*, mesocarp; *p*, hairs; *ct*, transverse cells; *t*, tubular cells; *sc*, hardened cells of the mesocarp; *bb'*, brown seed-coat envelopes; *c*, pigment; *fv*, fibro-vascular ducts; *h*, hyaline layer; *ap*, proteid

in the tissues of these various ground grains, their close resemblance is confusing. The size and characteristic grouping of the starch granules furnish by far the most ready means of distinguishing them.

Barley, even when ground fine, contains more of the seed-coat fragments and bits of bran than does wheat or rye, and the tissue elements are generally larger and better defined.

Wheat flour, according to Vogl, is readily distinguished from the flour of other grains by rubbing the cover-glass back and forth over the

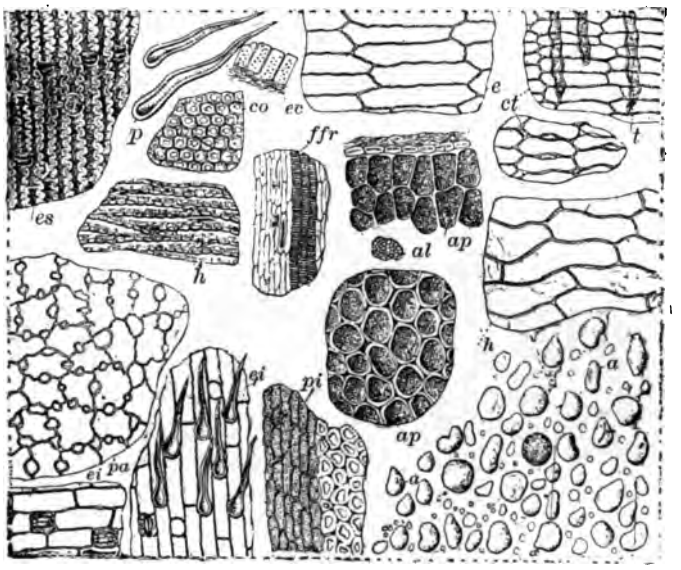


FIG. 59.—Barley Meal under the Microscope. *es*, exterior epidermis of the fruit; *pa*, parenchyma of the fruit; *h*, fibrous hypodermis; *ei*, interior epidermis of the fruit; *e'*, epicarp; *ct*, transverse cells; *t*, tubular cells; *ap*, proteid layer; *h*, hyaline envelope; *al*, aleurone; *pi*, masses of pigment; *a*, starch; *ffv*, fibro-vascular ducts. (After Villiers and Collin.)

water-mounted sample and observing the thread-like formations of gluten characteristic of the wheat. These are stained red with cochineal, are not at all apparent in the flour of rye, and hardly at all in that of other grains.

In order to bring out in a striking manner the elements of the various flours, Vogl* recommends the following mode of procedure. About 2 grams of the meal are placed in a dish and stained with an alcoholic solution of naphthylene blue. A pinch is removed to a microscope slide

* Die wichtigsten vegetabilischen, Nahrungs u. Genussmitteln, p. 17.

and examined in a drop of sassafras oil, or other analogous ethereal oil, or guiacol. The elements of the various layers and of the bran fragments are clearly distinguished by their bright-blue or blue-violet color, the

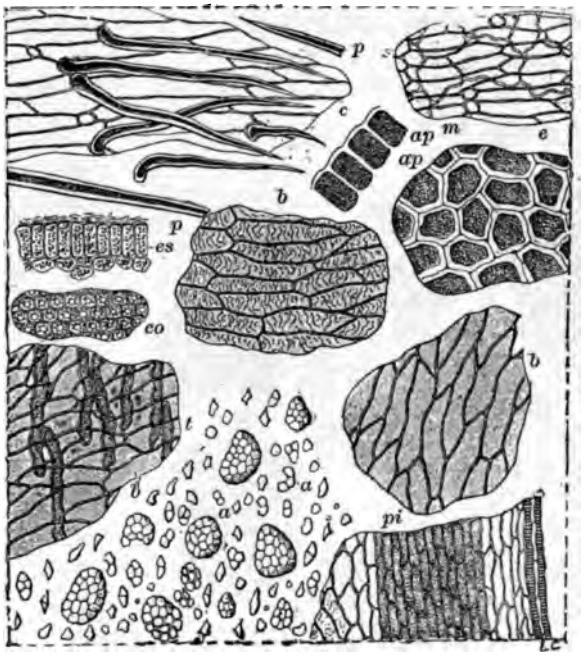


FIG. 60.—Oatmeal under the Microscope. *e*, epicarp; *p*, hairs; *b*, tegument of the grain; *ap*, proteid layer; *t*, tubular cells; *es*, envelope around embryo sheath; *co*, cotyledons; *pi*, masses of pigment; *a*, starch. (After Villiers and Collin.)

aleurone and germ tissues are stained pale blue, while the starch cells and starch grains remain colorless.

understood to mean the raised or leavened product, rendered light and porous by the aid of gas, which is generated either before or during baking. Commonly the gas employed is carbon dioxide, generated either by the fermentative action of yeast on the sugar of the dough, yielding both alcohol and gas, or by the agency of baking chemicals mixed with the dough, whereby an alkaline bicarbonate is decomposed by the action of an acid to produce the gas. Again, the gas may consist wholly or in

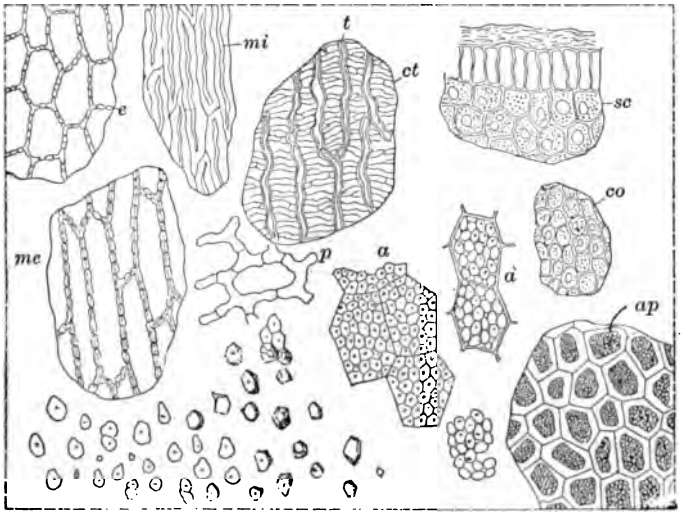


FIG. 61.—Corn Meal under the Microscope. *e*, epicarp; *me*, outer portion of mesocarp; *mi*, inner portion of mesocarp; *p*, lacunal parenchyma; *ct*, transverse cells; *t*, tubular cells; *sc*, scutellum with surrounding envelope; *ap*, proteid layer; *a*, starch, both in granules and masses; *co*, cotyledons. (After Villiers and Collin.)

part of ammonia, yielded by the vaporization during baking of ammonium carbonate mixed with the dough; and finally, the expansion during baking of the air itself confined in the dough may be the leavening agent, as in the case of cake and pastry.

Wheat flour is of chief value for bread on account of its high content of gluten, in which other cereals are lacking. In the preparation of ordinary white bread, the flour is mixed with water or milk, salt, and yeast, the materials are mingled thoroughly by kneading and allowed to remain for some time in a warm place, during which, by the vinous fermentation induced by the yeast, the mass "rises" or forms a light sponge, due to the action of the gas on the glutinous dough.

During the subsequent process of baking, which should take place at

a temperature between 230° and 260° C., further expansion ensues, much of the water is driven off, and the porous mass sets to form the loaf, the outside of which is converted into a brown crust, due to the caramelizing of the dextrin and sugar into which the starch of the outer layers is converted. Among other changes that take place in the interior or "crumb" during baking are (1) the partial breaking up of the starch grains, which, however, largely retain their identity, though in some degree distorted in shape; (2) somewhat obscure changes in the character of the proteids; and (3) partial oxidation of the oil or fat.

The standard for judging the quality of commercial bread may well be based on that of the best home-baked family loaf. The well-made loaf should possess an agreeable odor, and a sweet, nutty flavor, entirely free from mustiness. It should be well "raised," with a good crumbling fracture; it should not be tough or soggy on the one hand (due to under-raising), nor extremely dry and spongy on the other (indicative of over-raising). Over-raising, moreover, produces sourness, due to advanced lactic fermentation.

Composition of Bread.—The following analyses made in the U. S. Bureau of Chemistry of common varieties of bread were summarized from Bulletin 13, part 9, averages of a number of analyses being given in each case:

	No. of Analyses.	Moisture.	Proteids, N×6.25.	Proteids, N×5.70.	Ether Extract.
Vienna bread	10	38.71	8.87	8.09	1.06
Home-made bread	2	33.02	7.94	7.24	1.95
Graham bread	9	34.80	8.93	8.15	2.03
Rye bread	7	33.42	8.63	7.88	0.66
Miscellaneous bread	9	34.41	7.60	6.93	1.48
Biscuits or crackers	48	7.13	10.34	9.43	8.67
Rolls	11	27.98	8.20	7.48	3.41

physical characteristics of the loaf, its color, taste, odor, porosity, etc. In addition to such data, determination of moisture, ash, and acidity will usually suffice to enable the analyst to pass judgment on its wholesomeness. The following summary gives such analytical data on upwards of fifty samples of bread, purchased from cheaper bakeries and stores, and examined in the author's laboratory.

BREAD.

Kind of Bread.	No. of Analyses.	Weight of Loaf in Grams.	Water, Per Cent.	Per Cent Ash in Terms of Solids.	Acidity.*
White	44				
Maximum	..	653	45.20	1.83	6.2
Minimum	..	126	33.00	0.60	1.3
Mean	..	430	40.72	0.85	2.6
Graham	7				
Maximum	..	500	45.20	1.55	4.2
Minimum	..	367	40.10	0.96	2.1
Mean	..	420	41.50	1.26	3.5
Whole wheat	1	507	45.10	1.20	
Diabetic	1	445	47.00	2.20	
Muffins	1	194	48.20	1.15	1.7
Rye	1	1291	47.15	2.13	10.0
"Black"	1	550	47.00	2.20	
German with seeds	1	417	42.30	0.95	
Brown	1	500	48.10	3.50	
"Knackerbrod"	1	110	8.00	1.94	

* Cubic centimeters of tenth-normal soda required to neutralize 10 grams of the fresh bread.

Water in Bread.—The amount of water is of considerable importance, and, in the best bread, varies from 33 to 40 per cent. A larger content of water than 40% should be considered objectionable in a white bread, both on the ground of acting as a make weight, and because a large excess of moisture tends to cause the growth of mold.

Acidity of Bread.—The degree of sourness of a sample of bread is one of the most important indications as to its quality, and is most readily obtained by rubbing up in water, by means of a pestle, 10 grams of the "crumb," and titrating with tenth-normal alkali, using phenolphthalein as an indicator. To neutralize the acidity of 10 grams of the normally sweet loaf, an average of 2 cc. of the standard alkali solution is required, corresponding to 0.72 gram of lactic acid per loaf of an average weight of 400 grams. The loaf exhibiting the maximum sourness or acidity in the above table required 10 cc. of standard alkali per 10 grams of bread, corresponding to 11.61 grams lactic acid in the loaf of 1,291 grams.

Fat in Bread.—It is well known that the results of fat or ether extract as obtained by the ordinary method and expressed in most bread analyses are too low, being considerably less than the combined fat of the materials entering into its composition. This is probably due to the fact that during baking the fat particles are incrusted with insoluble matter, which protects them from the subsequent action of the ether. It is further claimed by some that the partial oxidation of the fat during baking has something to do with the low results. No perfectly satisfactory improvement over the regular Soxhlet method for fat extraction in bread has been discovered, and therefore this method, as described elsewhere, is recommended.

Adulteration of Bread.—The fraudulent addition of inert foreign ingredients to bread is almost never practiced, and is mainly of historic interest. Gypsum, chalk, bone ash, and various other minerals have been mentioned as possible adulterants, but the amount of any of these materials necessary to add for purposes of profit could scarcely be present without very apparent injury to the quality of the bread. Their presence in any considerable degree would be apparent in the abnormally high ash content of the bread.

The employment of alum to "improve" inferior or unsound flour has already been referred to, and, for the same purpose, sulphate of copper in small quantities is also said to have been used, enabling the making of bread of fairly good appearance from flour that was distinctly damaged.

Alum in Bread * is tested for by a modification of the logwood process described on page 237 as follows: 5 cc. of the logwood tincture and 5 cc. of the saturated ammonium carbonate solution are diluted to 100 cc., and the freshly prepared mixture poured over about 10 grams of the



Copper Salts in Bread are detected in the ash by the same method as that used for canned goods (p. 704).

Cake and Similar Preparations.—These differ from bread chiefly by the addition of considerable sugar, butter, spices, and other flavoring materials. In gingerbread, molasses is used as an important ingredient besides ginger. The adulterants of molasses, such as glucose, salts of tin, etc., would thus sometimes occur in gingerbread. In fact stannous chlorid has been found in ginger cakes.*

The following analyses of a few typical varieties of cakes are selected from Bulletin 13 of the Bureau of Chemistry:

	Moisture.	Proteids, N×6.25.	Proteids, N×5.70.	Ether Extract.	Crude Fiber.
Doughnuts	21.61	6.73	6.14	19.33	0.60
Ginger snaps	4.86	6.06	5.53	15.44	0.79
Fruit cake	24.47	4.56	4.16	12.35	
Gingerbread	21.49	6.25	5.70	8.42	0.90
Cup cakes	14.81	5.24	4.78	15.56	0.27
Macaroons	8.06	6.67	6.08	12.97	1.41
Jumbles	13.34	7.62	6.95	14.79	1.04

	Ash.	Salt.	Sugar.	Carbohydrates other than Fiber and Sugar.	Calculated Calories.
Doughnuts	0.40	0.03	1.28	50.64	5529
Ginger snaps	1.82	0.47	28.66	24.90	4971
Fruit cake					
Gingerbread	1.55	0.28	9.48	52.46	4757
Cup cakes	1.21	0.34	32.48	30.89	5073
Macaroons	0.82	0.07	58.77	10.89	4835
Jumbles	0.97	0.39	16.60	46.31	5133

LEAVENING MATERIALS.

YEAST.—The yeast plant is a fungus of the genus Saccharomyces, widely distributed through the vegetable kingdom and in the air. It is capable of rapid growth by the multiplication of its cells when present in a favorable medium, such as malt wort, and with propitious conditions of temperature, moisture, etc. Under such conditions, it forms a yellowish, viscous, frothy substance, the chief value of which, in the liquor industry, is the production of alcohol, while for bread-making, as a result of the same kind of fermentation, the end desired is the leavening of the doughy mass by the carbon dioxide liberated.

* See U. S Dept. of Agric., Bur. of Chem , Bul. 13, p. 1369.

A vigorous, pure yeast which will "raise" quickly is a great preventive against sour bread, for not only is it comparatively free from the germs and products of lactic acid fermentation, but by doing its work quickly it enables the baker to check the fermentation or raising process before the lactic acid or sour decomposition is far advanced.

Yeast most commonly used in bread-making is of the so-called "compressed" variety. The use of compressed yeast is almost universal for domestic purposes, and is more or less common in bakeries. A small amount of brewers' yeast in liquid form from beer wort is used, especially in the immediate neighborhood of breweries, and dry yeasts are used to some extent in localities so remote that fresh compressed yeast cannot readily be obtained.

Compressed Yeast is a product of distilleries where malt and raw grain are fermented for spirits. Most of it comes from whisky wort, and some from the worts used in the manufacture of gin and other distilled liquors. Little if any of the commercial compressed yeast is made from beer wort yeast.

In the manufacture of compressed yeast, the yeast floating on the top of the wort is separated by skimming, while that settling to the bottom is removed by running the wort into shallow settling trays. Top yeast is considered more desirable than bottom yeast for bread-making. The separated yeast is washed in cold water, and impurities are removed, either by sieving through silk or wire sieves, or by fractional precipitation while washing. The yeast, with or without the addition of starch, is finally pressed in bags in hydraulic presses, after which it is cut into cakes, packed in tin-foil, and kept in cold storage till distributed for use.

Such yeast should be used when fresh, as it readily decomposes and



in leavening as does compressed or brewers' yeast, but when once it begins to act it is quite as efficacious.

Composition of Yeast.—The following is the result of the analysis of under-fermentation yeast, after drying, by Nägele and Loew:

Cellulose and mucilage	37
Albuminoids (mycroprotein, etc.)	36
" soluble in alcohol	9
Peptones (precipitable by subacetate of lead)	2
Fat	5
Extractive matters (leucin, glycerin, etc.)	4
Ash	7
	100

Lintner gives the following average analyses of the ash of three samples of yeast, analyzed by him:

Silica	1.34
Iron (Fe_2O_3)	0.50
Lime (CaO)	5.47
Sulphuric anhydride (SO_3)	0.56
Magnesin (MgO)	6.12
Phosphoric anhydride (P_2O_5)	50.60
Potash (K_2O) and a little soda	33.49
	98.08

Matthews and Scott give the following as the ash composition of yeast:

Potassium phosphate	78.5
Magnesium phosphate	13.3
Calcium phosphate	6.8
Silica, alumina, etc.	1.4
	100.0

Microscopical Examination of Yeast.—Mix a bit of the yeast in water on the glass slip till a milky fluid is formed, and stir in a drop of a very weak anilin dye solution, such as methyl violet, eosin, or fuch-

sin.* Put on the cover-glass, and examine under the microscope. Living, active cells resist the stain, if the latter is dilute enough, and appear colorless or nearly so, while the decayed and lifeless cells are stained, and can easily be distinguished by their color. Yeast cells are circular or oval in shape, and vary from 0.007 to 0.009 mm. in diameter. They are sometimes isolated, and sometimes grouped in colonies; each cell has an outer, mucilaginous coating or envelope. The interior, granular mass or substance of the cell is the protoplasm, and within the protoplasm are frequently seen one or more circular empty spaces known as vacuoles.

Yeast cells multiply by the process of budding. The decadence of yeast cells is marked by the increased size of vacuole, and by the thickening of the cell wall.



a *b*

FIG. 62.—Sprouting Yeast-cells (*Saccharomyces cerevisiæ*). (*a*, after Lürssen; *b*, after Hansen.)

Yeast-testing.—*Available Carbon Dioxide.*—The value of yeast in bread-making depends on the amount of carbon dioxide which it is capable of generating under given circumstances, hence the available carbon dioxide is the chief factor in gauging a yeast. There are various methods of determination, (1) either by measuring the volume of gas set free by the action of a weighed quantity of yeast in a sugar solution of known strength kept for a fixed time at a fixed temperature (say 30°), or (2)



this flask, and dissolve 4.5 grams of the above sugar phosphate mixture. Finally add 1 gram of the yeast to be tested, stir it well till there are no lumps, and cork the flask. Carefully weigh on a delicate balance the flask with its contents, and immerse in a water-bath at 30° C., keeping it at that temperature for six hours. At the end of this time, remove the flask from the bath, and immediately immerse in cold water to cool the contents. Remove the rubber tube with the glass plug, and by suction draw out the remaining carbon dioxide. Replace the plug, and having carefully wiped off the flask, again weigh. The loss in weight is due to carbon dioxid set free by the fermentation of the yeast.

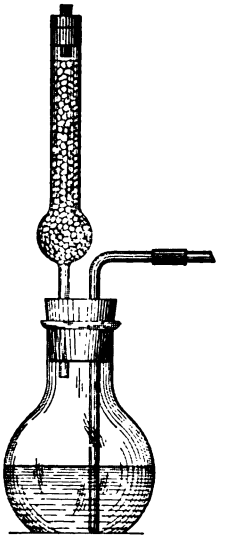


FIG. 63.—Apparatus for Determining Leavening Power of Yeast.

Starch in Compressed Yeast.—The addition of potato starch to yeast before pressing has long been customary, on the grounds that the starch acted as a drier, producing a much cleaner product, and one that could be more readily and intimately mingled with the materials of the bread, besides enhancing the keeping qualities of the yeast, especially in warm weather. The best grades of compressed yeast contain about 5% of starch, but some are found with 50% and even more. Undoubtedly the larger amounts are added as a make weight.

The question has frequently been raised whether, with improved methods of manufacture, whereby yeast could be produced comparatively free from slime, and thus capable of pressure without the admixture of starch, the use of the latter should not be considered as an adulterant. Some of the compressed yeast on the market is free from starch, and its makers claim that this is the only absolutely pure variety, while the presence of starch should be distinctly regarded as a violation of the section of the food law which forbids the use of a cheaper or inferior ingredient.

With the marked advantages possessed by a yeast containing starch, it is difficult to see why it should be considered as an adulterant, especially if present in moderate quantities. Briant claims that the admixture of starch up to 5% increases rather than decreases the actual content of yeast, in that the starch abstracts moisture from the yeast cells themselves, the proportion of water being much smaller, and that of the yeast larger in the starch mixed substance.

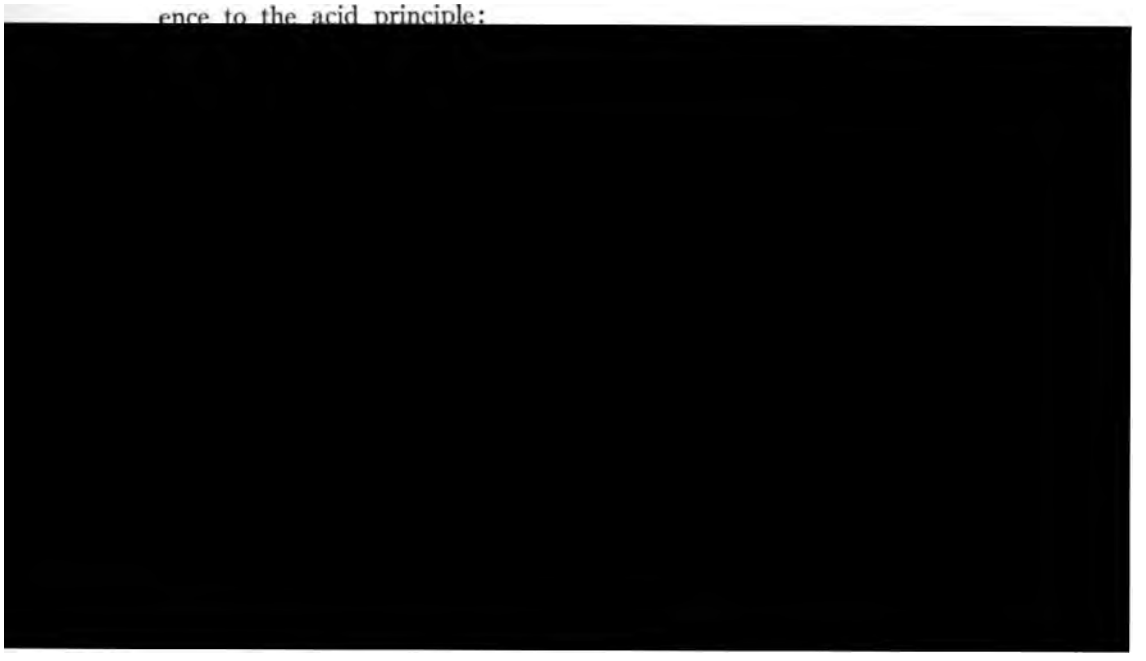
In the absence of a legal standard for starch in yeast, it is difficult to see how complaints could be maintained under the general food laws of most states, without condemning the use of starch altogether.

Jago suggests 20% as the limit for starch in yeast, beyond which it should be considered as an adulterant.

CHEMICAL LEAVENING MATERIALS.

Under this heading are included the various ingredients that enter into the mixtures commonly known as "baking powders" which have no food value in themselves, but are, strictly speaking, instruments or tools that by purely chemical reactions bring about, under certain conditions, the comparatively quick liberation of gas and the consequent aeration of biscuit, bread, and cake.

Baking Powders and their Classification.—Formerly the housewife was accustomed to measure out in proper proportion a mixture of sour milk, or cream of tartar, with saleratus to produce quick aeration of bread. The modern baking powder is a natural outgrowth of the former practice, and has almost wholly displaced it, producing, as it does, a mixture ready for immediate use of an acid and an alkaline constituent in proper proportion for chemical combination to form the gas. A third ingredient is, however, necessary in the commercial powder to check deterioration, viz., a dry, inert material, which by absorbing moisture prevents the premature chemical action between the reagents. Starch is nearly always used for this purpose, though sugar of milk has a limited use. The alkaline principle of nearly all baking powders is bicarbonate of soda, or saleratus. Baking powders are divided naturally into three main classes, with reference to the acid principle:



(2) *Phosphate Powders*, in which calcium acid phosphate is the acid principle:

$$\underset{\text{Calcium acid phosphate}}{\overset{234}{CaH_4(PO_4)_2}} + \underset{\text{Sodium bicarbonate}}{\overset{168}{2NaHCO_3}} = \underset{\text{Calcium monohydrogen phosphate}}{\overset{136}{CaHPO_4}} + \underset{\text{Disodium phosphate}}{\overset{142}{Na_2HPO_4}} + \underset{\text{Carbon dioxide}}{\overset{88}{2CO_2}} + \underset{\text{Water}}{\overset{36}{2H_2O}}$$

(3) "*Alum Powders*," wherein the acidity is due wholly or in part to sulphate of aluminum as it occurs in potash or ammonia alum, or in the mixed sulphates of aluminum and sodium.*

Assuming burnt potash alum as the substance used, the reaction would be as follows:

$$\underset{\text{Burnt potash alum}}{\overset{1020}{K_2Al_2(SO_4)_4}} + \underset{\text{Sodium bicarbonate}}{\overset{504}{6NaHCO_3}} = \underset{\text{Aluminum hydrate}}{\overset{156}{Al_2(OH)_6}} + \underset{\text{Sodium sulphate}}{\overset{426}{3Na_2SO_4}} + \underset{\text{Potassium sulphate}}{\overset{174}{K_2SO_4}} + \underset{\text{Carbon dioxide}}{\overset{264}{6CO_2}}$$

Naturally many baking powders of complex composition are met with, embodying various mixtures of the above classes.

Composition of Various Baking Powders.—Following are analyses of typical baking powders of the above classes: †

1. *Cream of Tartar Baking Powder:*

Total carbon dioxide, CO_2	13.21
Sodium oxide, Na_2O	13.58
Potassium oxide, K_2O	14.93
Calcium oxide, CaO	.18
Tartaric acid, $C_4H_4O_6$	41.60
Sulphuric acid, SO_3	.10
Starch	7.42
Water of combination and association by difference	8.98
	100.00

Available carbon dioxide 12.58%.

* It is probable that very little ammonia or potash alum is actually used at present in this class of powders. A product largely used is known in the trade as C. T. S. (cream of tartar substitute) and is a calcined double sulphate of aluminum and sodium.

† Div. of Chem., Bul. 13, part 5, pp. 600, 604, and 606.

2. *Phosphate Baking Powder:*

Total carbon dioxide, CO_2	13.47
Sodium oxide, Na_2O	12.66
Potassium oxide, K_2O	.31
Calcium oxide, CaO	10.27
Phosphoric acid, P_2O_5	21.83
Starch	26.41
Water of combination and association by difference	15.05
	100.00

Available carbon dioxide 12.86%.

3. *Alum Baking Powder:*

Total carbon dioxide, CO_2	9.45
Sodium oxide, Na_2O	9.52
Aluminum oxide, Al_2O_3	3.73
Ammonia, NH_3	1.07
Sulphuric acid, SO_3	10.71
Starch	43.25
Water of combination and association by difference	22.27
	100.00

Available carbon dioxide 8.10%.

Mixed Powders:

Total carbon dioxide, CO_2	10.68
Sodium oxide, Na_2O	14.04
Calcium oxide, CaO	1.29
Aluminum oxide, Al_2O_3	[redacted]

as baking powder. Unless a specific law forbids the use of a particular ingredient or class of ingredients, or in some manner regulates the labelling of the package, no baking powder of any kind can be considered adulterated under the general food law, unless it can be proved to be injurious to health, or unless it contain inert and useless mineral matter.

As a matter of fact, the residue left in the bread by all classes of baking powder consists of one or more drugs recognized in the Pharmacopœia, all of which *in large quantity* exercise well-marked toxic effects on the human system. Artificial digestion experiments, and physiological tests on the lower animals, using excessive doses of any of the above drugs, do not show the effect of the every-day use of baking powder in bread on the human system, and only a systematic examination of the effect of such use on large numbers of people can prove conclusively whether or not any one class of baking powders is harmful, and hence whether or not it should be classed as adulterated. Aside from the question of the harmfulness of the acid ingredients employed in baking powder, which is the subject of much controversy among rival manufacturers, there can be no doubt that such inert make weight substances as calcium sulphate, or terra alba, or clay, which are entirely useless, and lower the strength, quality, and purity of the powder, are to be considered in the light of adulterants.

Cream of Tartar—*Its Nature and Adulteration.*—Cream of tartar, or potassium bitartrate ($KH_5C_4O_6$), is the purified product obtained by the recrystallization of the crude argols or lees deposited in the interior of wine casks.

The lees, or argols, consist chiefly of crude potassium bitartrate, which is present in the juice of the grape, but is insoluble in the alcohol formed in the fermentation, and is hence deposited. If, for the clarification of the wine, such substances as gypsum or plaster of Paris are used, tartrate of calcium will be found mixed with the bitartrate of potassium in the lees. Hence it is that calcium tartrate is sometimes found in commercial cream of tartar.

Potassium bitartrate is insoluble in alcohol, sparingly soluble in cold, and readily soluble in hot water.

Allen * states that when the calcium tartrate is present in excess of 10%, it should undoubtedly be considered as an adulterant.

Other common adulterants of cream of tartar are calcium acid phosphate, gypsum, or plaster of Paris, starch, and alum.

* Analyst, V, 114.

Chemical Analysis of Baking Chemicals.—The degree of purity of cream of tartar is best determined by weighing out 0.188 gram of the sample, dissolving in hot water, and titrating with tenth-normal sodium hydroxide, using phenolphthalein as an indicator. If the article is pure, exactly 10 cc. of the standard alkali will be required for the titration. All the above-named adulterants, with the exception of alum, are either insoluble, or sparingly soluble in hot water, and will indicate the impurity of the sample even before titration. If the adulterant be alum, the sample would go into solution in the water, but the alum would be precipitated by the sodium hydroxide, the precipitate being, however, soluble in an excess of the alkali.

Sodium Bicarbonate on account of its cheapness is rarely adulterated, save by the occasional presence of common salt, an impurity incidental to its manufacture. The degree of purity of sodium bicarbonate is best ascertained by titration with standard acid, each cubic centimeter of tenth-normal acid being equivalent to 0.0084 gram of sodium bicarbonate.

DETERMINATION OF TOTAL CARBON DIOXIDE.

Reagents.—*Calcium Chloride.*—This can be obtained in granulated form in pellets of about the size of peas, specially prepared for moisture absorption.

*Soda Lime.**—To a kilogram of commercial sodium hydroxide, 500 to 600 cc. of water are added, and the mixture heated in an iron kettle to form a thin paste. While still hot, a kilogram of coarsely powdered quicklime is added, stirring with an iron rod. The lime is slaked, and the whole mass heats and steams up. No outside heat is necessary at this stage, but the mass is stirred and the lumps broken up. As soon as cool, place



absorbent. Types of these varieties are the Geissler and the Knorr apparatus

The Geissler Apparatus.—This consists of a flask *A*, having a ground neck *a*, and a flaring funnel-top *A'*. *B* is an elongated bulb, closed at the top by the hollow stopper *K*, and terminating below in the hollow stem *B'*, which is accurately ground at *b* to fit the neck *a*. Fused into the bulb *B* is the tube *C*, and within this is the small tube *D*, open at the top and communicating directly with the hollow stem *B'*. *gg* are openings between *B* and *C*.

E is a fine glass tube, passing from the bottom of the hollow stem *B'* and to the height of a small protuberance *e* in the bottom of the funnel *A'*, the construction being such that by turning the bulb and stem *BB'* in the neck *a* of the flask *A* the tube *E* may be opened or closed at the top. *H* is a side tube in the flask *A*, closed by the ground stopper *h*.

The bulb *B* and the tube *C* are filled with strong sulphuric acid nearly to the top of the tube *D*, by passing through the neck at the top, which is then closed by the stopper *K*.

About 0.5 gram of the dried sodium bicarbonate, or 1 gram of the baking powder, is introduced into the flask *A* through the neck *a* from a weighing-tube or otherwise, so that its exact weight is known. The stem *B'* is then inserted, and the funnel-top *A'* is nearly filled with the hydrochloric acid, the tube *e* being closed.

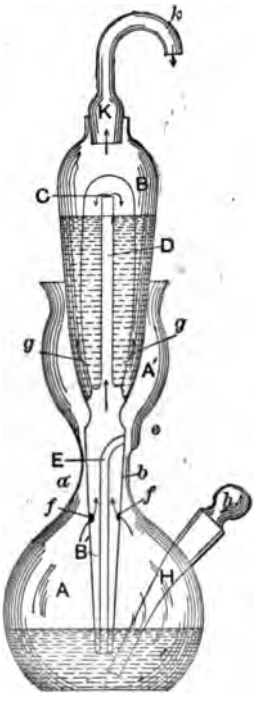


FIG. 64.—Geissler's CO_2 Apparatus or Alkalimeter.

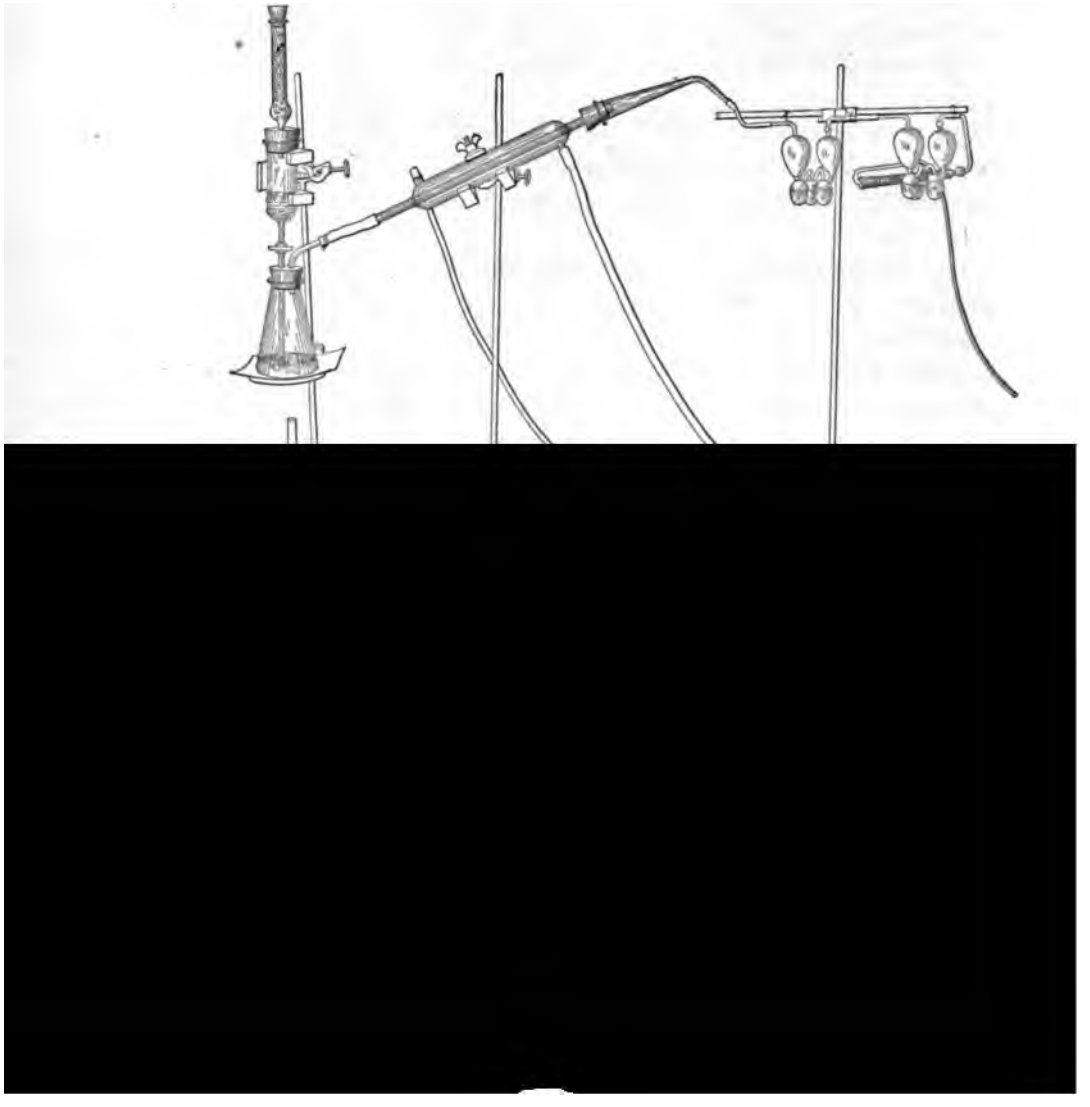
The entire apparatus is then weighed, after which the stem is turned to bring the protuberance *e* nearly opposite the tube *E*, uncovering it enough to allow the acid to pass slowly down the tube into the flask and upon the powder in the bottom of the flask. The carbon dioxide evolved passes through the opening *f* into the hollow stem *B'*, thence up through the tube *D*, and down and up (as indicated by the arrows) through the sulphuric acid, which absorbs the moisture. Finally the gas passes out through the tube *K*.

After the evolution of the gas has continued for two or three minutes, gentle heat is applied to the flask from a gas flame, and the solution is

brought to boiling, which is continued for a few minutes, during the latter portion of which the stopper *h* is removed, and the tubulure connected by rubber tubing with a system of two U tubes, one containing soda lime, and the other calcium chloride. The tube *k* is then connected with the aspirator, and a current of dried air is passed through the apparatus at the rate of about two bubbles per second, long enough to displace all the carbon dioxide. The rubber tubes are then disconnected, the stopper *K* is replaced, and the apparatus cooled to room temperature and weighed.

The *available* carbon dioxide in baking powder is determined in the same manner as above, by simply substituting freshly boiled, distilled water for the hydrochloric acid in the funnel-top *A'*.

The Knorr Apparatus (*Modified*).—The apparatus (Fig. 65) consists of (1) a flask, into which is introduced an accurately weighed amount of



chloride tube, which may if desired be replaced by a second sulphuric acid bulb. The potash absorption apparatus is accurately weighed before being connected up, and the funnel is nearly filled with the hydrochloric acid reagent, after which the soda lime tube is attached. The calcium chloride tube is connected by a rubber tube with the aspirator, and a current of cold water is allowed to run through the outer Liebig condenser-tube.

The stop-cock in the funnel-tube is first opened to allow the acid to slowly run into the flask, the flow being regulated to insure slow evolution of the gas.

The aspirator is then turned on so that about two bubbles of air per second pass through the apparatus, and gentle heat is applied to the flask by the gas flame, the solution within being brought to boiling, and the boiling continued for several minutes after the vapor has begun to gather in the condenser.

Prolonged boiling of the solution should be avoided, and in a series of tests the time of boiling should be precisely the same in all cases.

After removing the flame, the flask is allowed to cool, the aspiration being continued. The absorption-tube is then removed and weighed at room temperature, the increase in weight being due to the carbon dioxide.

The Available Carbonic Acid in Baking Powder is determined in the same manner as the total carbon dioxide, except that recently boiled, distilled water is substituted for the hydrochloric acid.

TARTARIC ACID.

Detection.*—It is frequently desirable to test a so-called "compound" cream of tartar, or a "cream of tartar substitute," or an adulterated sample made up largely of foreign ingredients, to see if *any* tartaric acid, free or combined, be present. The following test is applicable in presence of phosphates:

If the substance to be tested is found to be free from starch, mix a little of the dry powder in a test-tube with a bit of dry resorcin, add a few drops of concentrated sulphuric acid, and heat slowly. A rose-red color indicates tartaric acid or a tartrate, the color being discharged on dilution with water.

In case of baking powder, or a cream of tartar substitute containing

* Wolff, Rev. Chim. Analyt. et appl. 4 (1899), p. 2631.

starch, shake repeatedly from 3 to 5 grams of the sample with about 250 cc. of cold water in a large flask, allowing the insoluble portion to subside. Decant the solution through a filter, and evaporate the filtrate to dryness, after which test the dried residue or a portion thereof with resorcin and sulphuric acid as above described.

Determination of Total Tartaric Acid. — ***Modified Heidenhain Method.****—Applicable only in the absence of phosphates and salts of aluminum and calcium.

Into a shallow porcelain dish, 6 inches in diameter, weigh out 2 grams of the material and sufficient potassium carbonate to combine with all tartaric acid not in the form of potassium bitartrate. Mix thoroughly with 15 cc. of cold water, and add 5 cc. of 99% acetic acid. Stir for half a minute with a glass rod bent near the end. Add 100 cc. of 95% alcohol, stir violently for five minutes, and allow to settle at least thirty minutes. Filter on a Gooch crucible with a thin layer of paper pulp, and wash with 95% alcohol until 2 cc. of the filtrate do not change the color of litmus tincture diluted with water. Place the precipitate in a small casserole, dissolve in 50 cc. of hot water, and add standard fifth-normal potassium hydroxide solution, leaving it still strongly acid. Boil for one minute. Finish the titration, using phenolphthalein as indicator, and correct the reading by adding 0.2 cc. One cc. of fifth-normal potassium hydroxide solution is equivalent to 0.026406 gram tartaric anhydride ($C_4H_4O_6$), 0.03001 gram tartaric acid ($H_2C_4H_4O_6$), and 0.03763 gram potassium bitartrate ($KHC_4H_4O_6$).

The standard of the potassium hydroxide solution should be fixed by pure dry potassium bitartrate.

The accuracy of this method is indicated by the agreement of the percentages of potassium bitartrate in cream of tartar powders containing



a tared Gooch crucible, wash first with water until neutral, then once with alcohol, and finally with ether. Dry at 110° C. for four hours, cool, and weigh. Burn off the starch, and again weigh. The difference in the two weights indicates the weight of the starch. The purity of the starch is insured by examination with the microscope.

Acid Conversion Method.*—If the sample contains lime, mix 5 grams in a 500-cc. flask with 200 cc. of 3% hydrochloric acid, and let the mixture stand an hour with frequent shaking. Filter through a wetted 11-cm. filter, wash with water, and transfer the starch by a wash-bottle from the filter-paper back into the original flask, using 200 cc. of water.

If the sample be free from lime, weigh 5 grams directly into the 500-cc. flask with 200 cc. of water. In either case add 20 cc. of hydrochloric acid (specific gravity 1.125) and heat the flask in boiling water for 2½ hours, the flask being provided with a reflux condenser. Determine the dextrose, and from this the starch in the regular manner.

ALUMINUM SALTS.—Detection.†—(*a*) *In Baking Powder.*—Applicable in presence of phosphates. Burn to an ash about 2 grams of the sample in a platinum dish. Extract with boiling water and filter. Add to the filtrate sufficient ammonium chloride solution to produce a distinct odor of ammonia. A flocculent precipitate indicates aluminum.

In igniting, as above directed, sodium aluminate results from the more or less complete fusion. The reaction which occurs may be represented as follows:

$$Na_2Al_2O_4 + 2NH_4Cl + 4H_2O = Al_2(OH)_6 + 2NH_4OH + 2NaCl.$$

Sodium aluminate — Ammonium chloride — Aluminum hydroxide — Ammonia — Salt

If any phosphate of lime be present, it will be insoluble in the solution of the ash. If phosphate of sodium or potassium be present, it will go into solution, but will only precipitate out when an aluminum salt is also present on the addition of the ammonium chloride reagent.

(*b*) *In Cream of Tartar.*—Mix about 1 gram of the sample with an equal quantity of sodium carbonate, burn to an ash, and proceed as in the case of baking powder (*a*).

Determination of Alumina.—The above qualitative method with ammonium chloride may be made quantitative in presence of phosphates as follows: After carrying out the qualitative method as above directed, filter off the final precipitate, dissolve it in nitric acid, and test it for phos-

* U. S. Dept. of Agric., Bur. of Chem., Bul. 65, p. 105.

† Leach, 31st An. Rep. Mass. State Board of Health, 1899, p. 638.

phate with ammonium molybdate. If phosphates are found absent, proceed as before with a weighed amount of the sample and wash, ignite, and weigh the residue as Al_2O_3.

If phosphate is found present in the ammonium chloride precipitate, proceed as before, igniting and weighing the total residue. Then determine the P_2O_5 in the latter and subtract from the total. The difference will be the Al_2O_3.

Determination of Lime.—5 grams of the sample are treated in a 500-cc. graduated flask with 50 cc. of water and 25 cc. of concentrated hydrochloric acid. Add water to the mark, shake, and allow the starch to settle. Decant through a dry filter, and to 50 cc. of the filtrate add ammonia nearly to neutralization, and acidify with acetic acid. Then add ammonium acetate and boil. Filter, if necessary, and precipitate the lime with an excess of ammonium oxalate. Filter, wash, and ignite over a blast-lamp. Weigh as CaO.

Determination of Potash and Soda.*—Weigh out 5 grams into a platinum dish, and incinerate in a muffle at a low heat. The charred mass is well rubbed up in a mortar, then boiled fifteen minutes with about 200 cc. of water to which has been added a little hydrochloric acid. The whole is transferred to a 500-cc. flask, and, after cooling, made up to the mark and filtered. Of the filtered liquid 100 cc., representing 1 gram of the sample, are measured out, heated on the water-bath, and a slight excess of barium chloride added; then without filtering barium hydroxide is added in slight excess, the precipitate filtered off, and washed. To the filtrate is added a little ammonium hydroxide, and then ammonium carbonate until all the barium is precipitated. This precipitate is filtered and washed, the filtrate evapo-



Determination of Phosphoric Acid.—*Method of the A. O. A. C.**—Mix 5 grams of the material with 10 cc. of magnesium nitrate solution,† dry, ignite, and dissolve in hydrochloric acid. Take an aliquot part of the solution prepared above, corresponding to 0.25 gram, 0.50 gram, or 1 gram, neutralize with ammonia, and clear with a few drops of nitric acid. In case hydrochloric or sulphuric acid has been used as solvent, add about 15 grams of dry ammonium nitrate, or a solution containing that amount. To the hot solution add 50 cc. of molybdic solution‡ for every decigram of P_2O_5 that is present. Digest at about 65° for an hour, filter, and wash with cold water, or preferably ammonium nitrate solution.§ Test the filtrate for phosphoric acid by renewed digestion and addition of more molybdic solution. Dissolve the precipitate on the filter with ammonia and hot water and wash into a beaker to a bulk of not more than 100 cc. Nearly neutralize with hydrochloric acid, cool, and add magnesia mixture from a burette; add slowly (about 1 drop per second), stirring vigorously. After fifteen minutes add 30 cc. of ammonia solution of density 0.96. Let stand for some time; two hours is usually enough. Filter, wash with 2.5% NH_3 until practically free from chlorides, ignite to whiteness or to a grayish white, and weigh.

Determination of Sulphuric Acid.—*Provisional Method A. O. A. C.*‖—Boil 5 grams of the powder gently for one and one-half hours with a mixture of 300 cc. of water and 15 cc. of concentrated hydrochloric acid. Dilute to 500 cc., draw off an aliquot portion of 100 cc., dilute considerably, precipitate with barium chloride, filter through a Gooch crucible, ignite, and weigh. Direct solution of the material without burning the organic matter was proposed by Crampton.¶ The dextrose, formed by the action of the acid on the starch of baking-powders, does not interfere with the accuracy of the process.

Determination of Ammonia (present in the form of ammonia alum or ammonium carbonate). Mix 5 grams of the sample with 200 cc. of water, and add an excess of sodium hydroxide. Distill into standard acid, and determine the ammonia by titration.

* U. S. Dept. of Agric., Div. of Chem., Bul. 46, p. 12.

† Prepared as follows: Dissolve 80 grams calcined magnesia in nitric acid, avoiding an excess of acid, then add a little calcined magnesia in excess, boil, filter from the excess of magnesia, ferric oxide, etc., and dilute with water to 500 cc.

‡ Reagent No. 53.

§ Prepared by dissolving 100 grams of ammonium nitrate, Reagent No. 54, in 1 liter of water.

‖ U. S. Dept. of Agric., Bur. of Chem., Bul. 65, p. 107.

¶ U. S. Dept. of Agric., Div. of Chem., Bul. 13, part 5, p. 596.

SEMOLINA, MACARONI, AND EDIBLE PASTES.

Semolina is the coarse meal ground from certain varieties of hard or "durum" wheats, grown originally in Italy, Sicily, and Russia, but at present in France and certain parts of the United States and Canada. This hard wheat is high in gluten, and especially adapted for the preparation of macaroni and the various pastes. A peculiar process is employed in preparing the wheat, whereby the husk is removed by wetting, heating, grinding, and sifting, the resulting meal or *semolina*, being in the form of small, round, glazed granules.

The semolina thus prepared furnishes the basis of the Italian edible pastes, being mixed with warm water, kneaded, and molded into various forms and finally dried, either by pressure through holes in an iron plate, or otherwise.

Macaroni is the larger of the slender-tube or pipe-shaped products; vermicelli is the worm-shaped variety, produced when the holes in the plate are very small; *spaghetti* and *noodles* are terms applied to cord-like pastes intermediate in size between the others. A variety of Italian pastes or *pâtés* are made by rolling the kneaded semolina into thin sheets, and cutting out in shapes of animals, letters of the alphabet, etc.

The composition of some of these products is as follows:

	No. of Samples.	Water.	Protein.	Fat.	Total Carbohydrates.	Crude Fiber.	Ash.	Fuel Value per Pound. Cal's.
Semolina *	.....	10.50	11.96	0.60	75.79	0.50	0.65	
Macaroni †	11	10.3	13.4	0.9	74.1	....	1.3	1665
Noodles †	2	10.7	11.7	1.0	75.6	0.4	1.0	1665
Spaghetti †	3	10.6	12.1	0.4	76.3	0.4	0.6	1660
Vermicelli †	15	11.0	10.9	2.0	72.0	....	4.1	1625

celli. The wheat, softened by boiling, is shredded by passing through a peculiar machine, after which the biscuits are made by lightly putting together the threads and by final baking. The comparative composition of shredded wheat and of typical whole wheat is thus shown by Wiley:*

Constituents.	Shredded Biscuit. Per Cent.	Typical Wheat. Per Cent.
Moisture	10.57	10.60
Proteids	12.06	12.25
Ether extract	1.03	1.75
Ash	2.65	1.75
Crude fiber	2.58	2.40
Carbohydrates other than fiber	71.11	71.25

PREPARED CEREAL BREAKFAST FOODS.

The large number and variety of these preparations now on the market testify to the fact that the breakfast cereal forms a most important, as well as considerable, portion of our food supply. These foods are generally prepared from wheat, oats, and corn, and are, as a rule, remarkably pure and free from adulteration, though the food value of different varieties is often grossly misstated by their manufacturers. Formerly the breakfast food consisted entirely of the coarsely ground, generally decorticated, raw cereal grain, and required a long period of cooking to prepare it for use. At present many of the oat products, and to some extent also those of corn, rice, and wheat, are subjected to a more or less preliminary cooking and drying, whereby they are capable of being prepared for use in a much shorter time, and their keeping qualities are enhanced. The so-called rolled oats are prepared by softening the grains through steaming, after which they are crushed between rollers and afterwards dried. The steaming process is a typical one for various other cereals, though in some cases the heating consists in baking or kiln drying.

The effect of the preliminary cooking on the finished product varies somewhat according to whether dry or moist heat has been applied, and is chiefly noticeable in the altered character of the carbohydrates. In all cases the starch is rendered more soluble, whether by the conversion of a portion into dextrin and dextrose, or by a simple breaking down of the starch grains, as in the case of bread in baking.

In spite of the seemingly endless variety of the package cereals, they

* U. S. Dept. of Agric., Bur. of Chem., Bul. 13, p. 1337.

divide themselves as a matter of fact into a very few well-defined classes, the members of which differ but little from each other except in name.

First there are the raw cereal grains of the oat, wheat, and corn, prepared by simple crushing to various degrees of fineness, after decorticating; next comes the classes of partially cooked preparations of each of these grains, appearing in various forms of "flakes," "granules," "grits," etc., and again a class known as malted cereals, in which the moist, ground grain is mixed with malted barley, and, by controlling the temperature, a portion of the starch is converted to maltose and dextrin, after which the mixture is crushed between hot rollers and dried.

In the preparation of most of the corn breakfast products, such as samp and hominy, it is customary to remove the germ, which contains the oil and fat, lest the tendency of the latter to become rancid should result in the deterioration of the food. In wheat foods the germ is less often removed, and rarely, if ever, in oat preparations. The amount of fat found in the prepared cereal food as compared with that in the whole grain is of interest in this connection.

Composition of Some of the Common Breakfast Cereals.—The following analyses will serve to typify the various classes of these preparations as they appear on the market:

	Water.	Fat.	Protein.	Carbohydrates.				Ash.	Phosphorus.	Fuel Value per Gram. Calories.
				Total.	Soluble in Water.	Insoluble in Water	Crude Fiber.			
WHEAT.*										
Wheatena	6.65	2.28	14.17	75.62	3.9	70.50	1.22	1.28	0.363	4343
Pettijohn's breakfast food	9.51	1.45	10 56	76.96	2.8	72.15	2.01	1.52	0.231	4174
Farina	10.94	1.56	10.90	75.91	3.2	72.12	0.59	0.69	0.153	4051
Cracked wheat										

The methods of analysis employed for these preparations are the same as for ordinary cereals (p. 217), the sample being ground fine enough to pass through a 1-mm. sieve.

Prepared Foods for Infants and Invalids.—In dealing with the composition and analysis of this class of proprietary foods more than ordinary care is necessary, in view of the fact that one or another of these preparations are frequently prescribed for the exclusive diet of those whose very life may depend on the character and suitability of the food to the case in hand. Many of these foods do, as a matter of fact, honestly fulfill the claims of their manufacturers, but others fall far short of so doing, so that it is hardly safe to use them unless some intelligent idea of their composition can be gained. It is not, as a rule, within the province of the analyst to furnish an opinion regarding the adaptability of a certain food to the requirements of an infant or invalid, but rather to provide the necessary data whereon such an opinion may be intelligently based.

A simple statement of moisture, fat, protein, carbohydrates (by difference), and ash, which in the case of ordinary foods would often be sufficient, would be obviously inadequate in expressing the analysis of an infant food, since it is of much more vital importance than in other foods to know the solubility of the food itself, and, to as great an extent as possible, the character of the carbohydrates.

The chief ingredients of many of these preparations are wheat, or mixed cereals high in starch. Many of the foods are, according to the directions, to be used practically without cooking, but by simply mixing with milk or water, and, in some cases, bringing to the boiling-point. Hence the degree of conversion which the raw starch has undergone in the process of manufacture of the food should, if possible, be ascertained as a prime factor in judging of its character and adaptability to the needs of the young child and of the sick. Incidentally it should be said that few if any of the infant foods, even those whose high character has long been established by continued trial, conform very closely to the composition of woman's milk, which was long accepted as the true standard on which to base their efficiency. Hence it is no easy task to pass judgment on a particular food from its chemical composition alone without trial, nor is it right to unqualifiedly condemn in all cases food high in insoluble carbohydrates, since there are undoubtedly many instances in which such foods are successfully used.

Classification and Preparation of Infants' Foods.—These foods may for convenience be divided into two main classes, viz., *farinaceous foods*,

or those which are prepared wholly or chiefly from one or more cereal grains, and *lactated foods*, or those in which cow's milk forms the basis, but which may contain in addition thereto various other substances, such as cereals, sugars, etc.

The farinaceous foods, which are usually directed to be mixed with milk before using, may be further subdivided into (*a*) those that consist chiefly of unconverted starch, (*b*) those whose starch has been nearly all hydrolyzed to soluble form in the process of manufacture, and (*c*) those which contain much unconverted starch, but in addition thereto diastase or some other ferment, which, when the food is mixed with warm water or milk, is supposed to convert all the starch to soluble form.

The unconverted starch foods are nearly all made up of baked dry flour, chiefly that of wheat, but sometimes a mixture of cereals (as oats, barley, and wheat) and sometimes oats or barley alone. The baking breaks down to some extent the starch grains, as in the case of bread or crackers, but does not actually convert much of it to sugar.

The soluble farinaceous foods are usually prepared somewhat as follows: A mixture of ground wheat and barley malt (with sometimes a little wheat bran) is mixed with water to form a paste, and a little bicarbonate of potash added. The mixture is heated at 65° C. for sufficient time to convert the starch, after which it is exhausted with warm water, the extract being strained, and the filtrate evaporated to dryness to form the food. The sugars of such foods consist largely of maltose mixed with dextrin.

The farinaceous foods, which depend for the conversion of their starch on the method of cooking or heating before serving, are usually mixtures of wheat or other cereal flour with malt or pancreatic extract.

The milk foods are variously prepared, either by the simple desicca-

	Number of Analyses.	Moisture.	Fat by Petroleum Ether.	Loss to Alcohol.	Loss to Water.	Sum of Losses to Alcohol and Water.	Albuminoids, N×6.25.	Ash.
Farinaceous foods:								
Imperial granum	5	6.04	0.72	.....	3.94	3.94	13.77	0.49
Ridge's food	9	8.12	0.48	0.34	4.67	5.02	13.83	0.53
Mother's food	2	9.99	0.13	.....	.....	8.83	8.60	2.08
Robinson's barley	7	9.41	0.41	0.65	2.26	2.91	7.46	0.94
Mixed foods:								
Horlick's malted milk	9	2.55	1.41	.....	.....	63.87	14.00	3.57
Lactated food	12	5.77	0.48	28.24	4.27	32.90	10.01	2.57
Mellin's food	8	4.72	0.30	.....	.....	82.0	10.10	3.50
Nestlé's milk food	9	2.18	4.45	39.54	4.30	43.84	10.72	1.60
Reid & Carnrick's baby food	2	5.69	2.18	.....	.....	38.21	16.60	2.78

	Starch, Fiber, etc., by Difference.	Maltose.	Lactose.	Cane Sugar.	Remarks.
Farinaceous foods:					
Imperial granum	76.60	.....	.....	.....	Wheat starch
Ridge's food	72.01	.....	.....	.....	Wheat starch
Mother's food	69.24	.....	.....	3.00	Corn and wheat starch
Robinson's barley	78.66	.....	.....	.....	Barley starch
Mixed foods:					
Horlick's malted milk	15.68	49.00	.....	8.00	
Lactated food	47.72	.....	30.00	Trace	Wheat starch
Mellin's food	.....	50 to 60	.....	.....	
Nestlé's milk food	35.34	.....	8.96	36.34	Wheat starch
Reid & Carnrick's baby food	34.54	.....	30.00	8 to 9	

METHODS OF ANALYSIS.—The sample is prepared for analysis by grinding it sufficiently fine in a mortar or mill to pass through a 1-mm. sieve. Moisture, fat, ash, and nitrogen are determined as in the regular methods for cereals (pp. 217–219).

In determining loss of weight due to solubility of the sample in alcohol and water, proceed as follows:* The fat-free residue left in the Soxhlet apparatus, after extraction with ether or petroleum ether, is subjected to further extraction with 95% alcohol, till all soluble matter has been extracted. If 5 grams of the sample were originally taken for the fat extraction, this operation would require about five hours. Evaporate the alcoholic extract to dryness, and weigh the residue as in the case of the ether extract. Dry the residue left in the Soxhlet from the alcoholic extract, or a portion thereof, in a platinum dish over the water-bath, cool, and weigh. Transfer to a Gooch crucible, provided with asbestos

* McGill, Canada Inland Rev. Dept. Bul. 59.

and previously tared, a portion, the relation of which to the original weight taken is calculated from the moisture, ether, and alcohol extracts as previously determined. Pass through the contents in the Gooch by suction from 200 to 300 cc. of cold water at room temperature, dry the Gooch and its contents at 100° to constant weight, cool and weigh, thus determining the solubility of the sample in water.

According to McGill, five hours' extraction with alcohol under the above conditions removes all cane sugar, but probably not all the lactose, maltose, and dextrose, if a considerable quantity of these sugars is present. Water dissolves the dextrin and gum and such of the sugar as escapes solution in the alcohol, hence the sum of the alcohol and water extract is of value. In the calculation of the starch, fiber, etc., by difference, it should be borne in mind that the result is only approximate, by reason of the fact that the small amount of soluble albuminoids (which McGill states never exceeds 2½%) are reckoned in, hence a small error is introduced, which could be corrected, if considered worth while, by determining the amount of soluble albuminoids.

Separation of the Carbohydrates can be effected by Stone's method (pp. 227, 228), but a very satisfactory idea of the solubility of these foods, which is of chief importance, can be gained by the much simpler modified method of McGill, as described in the preceding paragraphs.

Cold-water Extract.—The equivalent of 10 grams of the moisture-free substance, finely ground, is weighed in a tared flask, and water added in several portions with gentle shaking till the contents of the flask weigh 110 grams. The flask is then corked and vigorously shaken at intervals during six or eight hours and allowed to stand over night. The supernatant liquid is then decanted into the large tubes of a centrifuge, and whirled till the sediment settles out. The comparatively clear liquid may

subjected to heating, or boiling with water or milk, to condemn them as containing much insoluble matter, without comparing the figures expressing results of the analyses of the raw foods, calculated to the water-free basis, with those obtained on analyzing the food after boiling or otherwise cooking with pure distilled water, for a length of time specified in the directions, and afterwards drying. It is possible that the presence in the food of diastase, or other ferment, may be depended on to hydrolyze a whole or a portion of the starch, and only by such comparison will this be shown.

Microscopical Examination of the food is of value in determining its general character, showing especially whether or not starch is present in its original form, or has been converted in whole or in part. The particular varieties of cereal grain employed are generally evident, as well as the presence and proportion of the different tissues of the grain.

REFERENCES ON CEREALS, VEGETABLES, LEGUMES, FRUITS, ETC.

ATWATER, HELEN W. Bread and the Principles of Bread Making. Farmer's Bul. 112.
—— Beans, Peas and other Legumes as Food. Farmer's Bul. 121.
BALLAND. Recherches sur les Blés, les Farines et le Pain. Paris, 1894.
BIGELOW, MUNSON, TOLMAN, and HOWARD. Fruits and Fruit Products. Bur. of Chem. Bul. 66.
BLASDALE, W. C. Some Chinese Vegetable Food Materials. Exp. Sta. Bul. 68.
CHITTENDEN, R. H., and OSBORNE, T. B. Proteids of the Corn Kernel. Am. Chem. Jour., XIII, No. 7, and XIV, No. 1.
GALT, H. The Microscopy of the Starches. London, 1900.
GOODFELLOW, J. The Dietetic Value of Bread. London, 1892.
GRIFFITHS, W. The Principal Starches Used as Food. Cirencester, 1892.
HANAUSEK, T. T. Die Nahrungs- und Genussmittel aus dem Pflanzenreiche. Kassel, 1884.
HARZ, C. O. Landw. Samenkunde. Berlin, 1885.
JAGO, W. Science and Art of Bread Making, Chemistry and Analyses of Wheat, Flour, etc. London, 1895.
KÖRNICKE, F., u. WERNER, H. Handbuch des Getreidebaues. Berlin, 1885.
KRUG, W. H. Analytical Methods for Carbohydrates as Applied to Foods, etc. Jour. Frank. Inst., 1902 (CLIV), 349-401.
LAWES and GILBERT. Composition of Wheat Grain, its Products in the Mill, and Bread.
MCGILL, A. Infants' and Invalids' Foods. Canada Inl. Rev. Dept. Bul. 59.
—— Cereal Breakfast Foods. Canada Inl. Rev. Dept. Bul. 84.
MAURIZIO, A. Getreide, Mehl und Brot. Berlin, 1903.
MOELLER, J. Mikroskopie der Nahrungs- und Genussmittel aus dem Pflanzenreiche. Berlin, 1886.
MOLISCH, H. Grundriss einer Histochemie der pflanzlichen Genussmittel. Jena, 1891.

MUNSON, L. S., and TOLMAN, L. M. Fruits and Fruit Products. Bur. of Chem. Bul. 65, Part XIII, page 74.
OSBORNE, T. B. Crystallized Vegetable Proteids. Am. Chem. Jour., XIV, No. 8.
—— Oat Proteids. Conn. Exp. Station, An. Reps., 1890, page 115; 1891, page 124.
—— The Proteose of Wheat. Am. Chem. Jour., XIX, No. 3.
—— Proteids of Rye. Jour. Am. Chem. Soc., 17, 429.
—— Proteids of Barley. Jour. Am. Chem. Soc., 17, 539.
—— Amounts and Properties of the Proteids of Maize. Jour. Am. Chem. Soc., 19, 525
OSBORNE, T. B., and CAMPBELL, G. F. Proteids of the Potato. Jour. Am. Chem Soc., 18, 575.
—— Proteids of the Pea. Jour. Am. Chem. Soc., 18, 583, and 20, 348.
OSBORNE, T. B., and VOORHEES, C. L. Proteids of Wheat. Jour. Am. Chem. Soc., 16, 524.
OSBORNE, T. B., and VOORHEES, C. G. Proteids of the Wheat Kernel. Am. Chem. Jour., XV, No. 6.
SCHIMPER, A. F. W. Anleitung z. mikrosk. Unters. der Nahrungs- und Genussmittel. Jena, 1886.
SHERMAN, H. C. Carbohydrates of Wheat. Jour. Am. Chem. Soc., 19, 1897, page 291.
SKINNER, R. P. Manufacture of Semolina and Macaroni. Bur. of Plant. Ind. Bul. 20.
SNYDER, H. Digestibility and Nutritive Value of Bread. Exp. Sta. Bul. 126.
SNYDER, FRISBY, and BRYANT. Losses in Boiling Vegetables. Exp. Sta. Bul. 43.
SNYDER, H., and VOORHEES, M. A. Studies on Bread and Bread-Making. Exp. Sta. Bul. 67.
STONE, W. E. Carbohydrates of Wheat, Maize, Flour, and Bread. Action of Enzymes on Starches. Exp. Sta. Bul. 24.
TSCHIRCH, A., und OESTERLE, O. Anatomischer Atlas der Pharmakognosie und Nahrungsmittelkunde. Leipzig, 1893.
VOGL, A. E. Verfälschungen und Verunreinigungen des Mehles und deren Nachweisung. Wien, 1880.
—— Die wichtigsten vegetabilischen Nahrungs- und Genussmittel. Wien u. Leipzig, 1899.
WANKLYN, J. A., and COOPER, W. J. Bread Analysis. London, 1886.
WILEY, H. W. Sweet Casava. Div. of Chem. Bul. 44.



Maine Exp. Station Bul. 54. Nuts as Food.
" " " " 55. Cereal Breakfast Foods.
" " " " 75. Analyses of Self-raising Flours, Pea Flours, Gluten Foods, Chestnuts, Malted Nuts.
" " " " 84. Cereal Breakfast Foods.
Minnesota Exp. Station Bul. 74. Digestibility and Food Value of Beans.
Penn. Dept. of Agriculture Bul. 10. Special Report on Prepared Foods for Infants and Invalids.
Wyoming Exp. Sta. Bul. 33. Composition of Prepared Cereal Foods.

REFERENCES ON LEAVENING MATERIALS.

CATLIN, CHAS. A. Baking Powders, with Special Reference to Phosphate Powders. Providence, 1899.
CRAMPTON, C. A. Baking Powders. Div. of Chem. Bul. 13, Pt. 5.
GREEN, J. R. Die Enzyme. Berlin.
—— The Soluble Ferments and Fermentation. 1901.
HANSEN, E. C. Practical Studies in Fermentation. London, 1896.
JORGENSEN, A. Die Mikro-organismen der Gärungsindustrie. Berlin.
—— Micro-organisms and Fermentation. London, 1900.
KLOCKER, A. Fermentation Organisms. 1903.
LINDNER, P. Mikroskopische Betriebskontrolle in den Gärungsgewerben. Berlin, 1895.
MCGILL, A. Baking Powders. Canada Inl. Rev. Dept. Buls. 10, 26, 68.
—— Cream of Tartar. Canada Inl. Rev. Dept. Buls. 12, 26, 71.
MATTHEWS, CHAS. G. Manual of Alcoholic Fermentation. London, 1901.
OPPENHEIMER, C. Trans. by Mitchell, C. A. Ferments and their Action. London, 1901.
PLUMMER, R. H. A. Chemical Changes and Products Resulting from Fermentation. London, 1903.
WINTON, A. L. Baking Powders. Bur. of Chem. Bul. 65, Part XV.

Conn. Exp. Sta. An. Rep., 1900, page 15.
Michigan Dairy and Food Commission, Bul. 2, page 12; Bul. 3, page 7.
Penn. Board of Agriculture An Report, 1897, page 166.
North Carolina Exp. Sta. Bul. 155.

CHAPTER X.

TEA, COFFEE, AND COCOA.

TEA.

Nature and Classification.—Tea consists of the prepared leaves or leaf buds of a plant belonging to the genus *Thea*, the *Thea chinensis* being the specific variety generally employed.

The best teas are made from young leaves only, the Chinese teas being classified with reference to the age and position of the leaf on the young shoot. Thus, the very choicest Chinese tea, rarely found outside of China, is prepared from the youngest or end leaves of the shoot, which are scarcely more than buds, and form the tea known as *pekoe tip*, or *flowery pekoe*. The next leaves are the *orange pekoe* and *pekoe*, which produce a very high grade of tea, while next in order as to age, size, and grade of leaf are the *souchong 1st* and *2d*, and the *congou*, producing teas called by the same names.

More than 50% of the tea consumed in the United States comes from China, and over 40% from Japan, the remainder being derived largely from India, Ceylon, and other East Indian ports.

In the manufacture of tea the fresh leaves, which are nearly 80% water, are rolled, withered by exposure to light, heat, and air, and finally dried

Composition of Tea.—König gives the following composition of fully developed tea leaves, being the mean of 50 to 70 analyses:

Water.	Nitrogenous Substances.	Theine.	Essential Oil.	Fat, Chlorophyl, and Wax.	Gum, Dextrin, etc.	Tannin.	Pectin, etc.	Crude Fiber.	Ash.
9.51	24.50	3.58	0.68	6.39	6.44	15.65	16.02	11.58	5.65

Though the nitrogenous substances of tea predominate in amount over any other class of constituents, yet, with the exception of theine or



FIG. 66.—*a*, Flowery Pekoe; *b*, Orange Pekoe; *c*, Pekoe; *d*, Souchong, 1st; *e*, Souchong, 2d; *f*, Congou; *a*, *b* (when mixed together), Pekoe; *a*, *b*, *c*, *d*, *e* (when mixed together), Pekoe Souchong. If there be another leaf below *f*, it is termed Bohea. At base of leaves are buds 1, 2, 3, 4, from which new shoots spring. (After Money.)

caffeine, they have been little studied. Theine, tannin, and essential oil give to the infusion of tea its chief characteristics.

Zollinski* gives the following summarized results of analyses of a number of the cheaper grades of Chinese black tea:

* Zeits. anal. Chem., 1898, 37, 365.

	Water.	Total Nitrogen.	Albuminoid and Amido-nitrogen.	Protein, N × 6.25.	Theine.	Ash.	Soluble Ash.	Insoluble Ash.
Maximum....	11.57	4.12	3.78	23.83	2.06	6.78	31.17	61.03
Minimum	9.96	3.76	3.37	21.06	1.14	4.79	28.13	57.74
Average.....	10.58	3.93	3.52	22.01	1.55	5.94	29.67	59.75

A very complete series of analyses of tea was made by Joseph F. Geissler in 1884,* from which the following summaries are taken:

		Number of Analyses.	Moisture.	Half-hour Extract.	Total Extract.	Insoluble Leaf.	Tannin.	Theine.	Total Ash.	Soluble Ash.	Insoluble Ash.	Ash Insol. in Acid.
Indian:	Maximum..	6	6.19	39.66	45.64	53.07	18.86	3.3	5.79	3.68	2.22	.296
	Minimum..	..	5.56	37.80	41.32	48.53	13.04	1.8	5.42	3.24	1.93	.137
	Average....	..	5.81	38.77	42.94	51.24	14.87	2.7	5.62	3.52	2.12	.178
Oolong:	Maximum..	13	6.88	44.02	48.87	53.15	20.07	3.50	6.11	3.71	3.17	.838
	Minimum..	..	5.09	34.10	40.6	44.8	11.93	1.15	5.44	2.60	1.84	.266
	Average....	..	5.89	37.88	43.32	50.7	16.38	2.32	5.81	3.2	2.68	.507
Congou:	Maximum..	11	9.15	32.14	37.06	63.85	13.89	2.87	6.48	3.52	3.86	1.31
	Minimum..	..	7.65	23.48	27.48	54.5	8.44	1.70	5.52	2.28	1.90	.32
	Average....	..	8.37	28.40	34.35	57.2	11.54	2.37	5.75	3.06	2.68	.425

Kenrick † gives the following averages of a series of analyses of tea made by him in 1891:

	Number of Analyses.	Substances Extracted by 10 Minutes' Infusion.			Moisture.	Soluble Ash.	Insoluble Ash.	Total Ash.	Ratio of Total Solids to Tannin.
		Total Solids.	Tannin.	Theine.					

	Oolong. Average of 50 Samples.	Japan.	Spent Black Tea.
Total ash	6.04	5.55	2.52
Soluble in water	3.44	3.60	0.28
Per cent soluble	57.00	64.55	11.11
COMPOSITION.			
Silica	11.30	9.30	27.75
Chlorine	1.53	1.60	0.79
Potash	37.46	41.63	
Soda	1.40	1.12	
Ferric oxide	1.80	1.12	16.00
Alumina	5.13	4.26	
Manganic oxide	2.10	1.30	
Lime	9.43	8.18	19.66
Magnesia	8.00	5.33	11.20
Phosphoric acid	12.27	16.62	15.80
Sulphuric acid	4.18	3.64	1.10
Carbonic acid	5.40	5.90	6.70
	100.00	100.00	99.00

Kozai * gives the following as the results of analyses made by him of Japanese teas:

	Unprepared Leaves.	Green Tea.	Black Tea.
Caffeine or theine	3.30	3.20	3.30
Ether extract	6.49	5.52	5.82
Hot-water extract	50.97	53.74	47.23
Tannin (as gallotannic acid)	12.91	10.64	4.89
Other nitrogen-free extract	27.86	31.43	35.39
Crude protein	37.33	37.43	38.90
Crude fiber	10.44	10.06	10.07
Ash	4.97	4.92	4.93
Albuminoid nitrogen	4.11	3.94	4.11
Caffeine nitrogen	0.96	0.93	0.96
Amido-nitrogen	0.91	1.13	1.16
Total nitrogen	5.97	5.99	6.22

PROXIMATE COMPONENTS AND ANALYTICAL METHODS.

Moisture.—From 2 to 5 grams are heated on a watch-glass or platinum dish at 100° in the air-oven to a constant weight (usually about three hours), calculating the moisture from the loss in weight.

Total Ash.—A weighed portion of the sample (that used for obtaining the moisture may be conveniently employed) is burnt to a white ash in a

* Bul. 7, Imperial College of Agriculture, Japan.

platinum dish at a low red heat, and the residue cooled and weighed. The total ash of pure tea should not be less than 4 nor more than 8 per cent.*

Soluble and Insoluble Ash.—The total ash, as obtained above, is transferred to a beaker with hot water and boiled for some time, after which it is poured upon a filter and the residue washed with hot water till all the soluble matter is extracted. The residue is then dried, ignited, cooled, and weighed, thus giving the amount of insoluble ash. The soluble ash is calculated by difference from the total and insoluble ash. At least 50% of the total ash should be soluble in water.*

Ash Insoluble in Acid.—The portion of the ash insoluble in water is washed upon a tared filter with hot 10% hydrochloric acid and further washed with the latter reagent till the acid-soluble matter is dissolved out, after which it is washed with water, dried, and weighed.

Total Nitrogen.—This is determined by the Gunning method on 1 to 2 grams of the sample.

Essential Oils.—Distill 100 grams of the tea with 800 cc. of water and shake out the distillate with several portions of ether. The residue from the combined ether extracts contains the volatile oil.

Ether Extract is determined by the regular method of continuous extraction, using dry alcohol-free ether or petroleum ether on 2 to 5 grams of the dried sample.

Crude Fiber is determined as on page 218 in the residue from the ether extract.

Theine or Caffeine ($C_8H_{10}N_4O_2$).—This alkaloid when pure exists in white silky needles. It is odorless and sparingly soluble in cold water, but more so in hot. It is less soluble in alcohol, and almost insoluble in ether. It readily dissolves in chloroform. It is present in tea, coffee,



if caffeine is present, a reddish-yellow or pink color is produced. After cooling, the residue is treated with a very little ammonia water applied on the point of a stirring-rod. In the presence of caffeine, a purple color (that of murexoin) is produced on application of the ammonia.

Determination of Theine or Caffeine.—*Modification of Stahlschmidt's Method.*—Six grams of finely powdered tea are boiled in a flask with several successive portions of water for ten minutes each, and the combined aqueous extracts thus obtained are made up to 600 cc. with water. Four grams of powdered lead acetate are added to the decoction, which is then boiled for ten minutes, using a reflux condenser, or making up the loss by occasional addition of water. The solution is then poured upon a dry filter, and 500 cc. of the filtrate, corresponding to 5 grams of the tea, are evaporated to about 50 cc. and enough sodium phosphate added to precipitate the remaining lead. The solution is then filtered, and the precipitate thoroughly washed, the filtrate and washings being evaporated to about 40 cc. Finally, the solution thus concentrated is extracted repeatedly with chloroform in a separatory funnel for at least four times, and the chloroform extract evaporated to dryness, leaving the caffeine, which is dried to constant weight at 75° and weighed.

Determination of Tannin.—*Procter's Modification of Lowenthal's Method.**

Reagents: (1) Potassium permanganate solution containing about 1.33 grams per liter.

(2) Tenth-normal oxalic acid solution (6.3 grams per liter).

(3) Indigo carmine solution, containing 6 grams indigo carmine (free from indigo blue) and 50 cc. concentrated sulphuric acid per liter.

(4) Gelatin solution, prepared by soaking 25 grams gelatin for an hour in a saturated sodium chloride solution, heating till the gelatin is dissolved, and making up to a liter after cooling.

(5) Mixture of 975 cc. saturated sodium chloride solution and 25 cc. concentrated sulphuric acid.

(6) Powdered kaolin.

Obtain the value of the potassium permanganate solution in terms of the tenth-normal oxalic acid solution.

* U. S. Dept. of Agric., Div. of Chem., Bul. 13, part 7, p. 890.

Boil 5 grams of the powdered tea for half an hour with 400 cc. of water, cool, transfer to a graduated 500-cc. flask, and make up to the mark. To 10 cc. of the infusion (filtered if not clear) add 25 cc. of the indigo carmine solution and about 750 cc. of water. Then add from a burette the potassium permanganate solution, a little at a time while stirring, till the color becomes light green, then cautiously drop by drop till the color changes to bright yellow,* or further to a faint pink at the rim. The volume in cubic centimeters of permanganate furnishes value *a* of the formula.

Mix 100 cc. of the clear infusion of tea with 50 cc. of gelatin solution, 100 cc. of salt acid solution, and 10 grams of kaolin, and shake several minutes in a corked flask. After settling, decant first the clear supernatant liquid through a filter, and finally bring the precipitate upon the filter. Mix 25 cc. of the filtrate (corresponding to 10 cc. of the original infusion) with 25 cc. of the indigo carmine solution, and about 750 cc. of water, and titrate with permanganate as before. The volume in cubic centimeters of permanganate used gives value *b*.

a = quantity of permanganate solution required to oxidize all oxidizable substances present.

b = quantity of permanganate solution required to oxidize substances other than tannin.

$\therefore a - b = c$, permanganate required for the tannin. Assuming that 0.04157 gram tannin (gallotannic acid) is equivalent to 0.063 gram oxalic acid, the tannin in the tea is readily calculated.

Method of Fletcher and Allen.†—This method depends upon the precipitation of the tannin and other astringent matters in tea infusion by lead acetate, the point of complete precipitation being indicated by an

[redacted]

solution, as above prepared, are measured into as many beakers, and each diluted to 100 cc. with boiling water. Two grams of powdered tea are boiled in 250 cc. of water, and varying quantities of this decoction are measured from a burette or pipette into the beakers containing the lead solution, the first beaker receiving, say, 12 cc., the second 15 cc., and the third 18 cc., in the case of black tea, and, with green tea, 8, 10, and 12 cc., respectively.

About 1 cc. of each of these trial quantities is removed from the various beakers by means of a pipette, passed through small filters, and tested with the ammoniacal ferricyanide indicator, the drops of filtered solution being allowed to fall directly on spots of the indicator, previously placed on a white slab or plate.

It is thus easy to ascertain the *approximate* amount of tea solution which it is necessary to add to produce a pink coloration with the indicator, so that by repeated tests, nearly the right amount may be added at once. If no coloration in a given case is produced when a drop of the filtrate from the solution in the beaker is allowed to fall on the drop of indicator solution, a little more of the tea decoction is added, and this process is repeated until the pink color is apparent.

It should be noted how much of the tea decoction is necessary to add to 100 cc. of pure water, that a drop of the solution may produce the pink coloration with the ferricyanide, and this amount should be subtracted from the amount of decoction found necessary to add to the known lead solution in the beaker. It was found by repeated experiment that 10 cc. of lead solution would precipitate 0.01 gram of pure gallotannic acid; hence, carrying out the process exactly as above described, 125 divided by the number of cubic centimeters of tea decoction required gives the percentage of tannin in the sample.*

Extract.—By this term is meant the total amount of water-soluble matter in tea, including such compounds as tannin, caffeine, albuminous matter, dextrin, gum, certain parts of the ash, etc.

The value of a tea from a food standpoint depends obviously upon the character and amount of the extract, rather than on the composition of the dry tea. The relative composition of the extract and of the insoluble leaves, as found by Eder, is given in the table on page 284.

Determination.—Extract successively 2 grams of the finely powdered tea with seven portions of boiling water, containing 50 cc. each, decanting the extract each time, and uniting the various portions into one.

* This process estimates the total astringent matter, all of which is counted in as tannin.

	Extract.	Insoluble Leaves.
	Per Cen	Per Cent.
Dry matter	40.	60.
Nitrogenous substances	12.	12.7
Theine	2.	
Tea oil	0.6	
Resin, chlorophyll, etc.	.....	7.2
Tannin	10.	
Extractives	12.	10.
Ash	1.7	2.3
Potash	0.94	0.29
Lime	0.04	0.58
Phosphoric anhydride	0.13	1.03
Silica	0.21	0.68

After boiling the total extract, it is passed through a tared filter, after which the insoluble residue is transferred to the filter and washed with boiling water. The filtrate and washings are made up to a noted volume, and an aliquot part is evaporated to dryness in a tared platinum dish to a constant weight.

Insoluble Leaf.—The tared filter with the washed residue from the extract, as above obtained, is dried in an air-oven at 110° and weighed. If the amount of insoluble leaf is above 60%, the presence of spent or exhausted leaves may be suspected.

ADULTERATION OF TEA.

Facing.—The most common form of tea adulteration, if such it may be called, is the practice of "facing" the dried leaves, or treating them with certain pigments and coloring materials to impart a bright color or gloss to the tea, thus causing an inferior grade to appear of better



mixture is then added while still hot, and the whole is stirred briskly till the desired color is imparted. The Chinese and Japanese do not face the tea which they themselves consume, but only that intended for export trade.

The microscope furnishes the most ready means of detecting turmeric and plumbago. The latter is detected by the bright glossy particles, evident when a thin section of the tea leaf is examined under the microscope.

Prussian blue and indigo are also evident by the microscopical appearance of the particles, detached by shaking the leaves in water. Prussian blue or ferric ferrocyanide is detected by the transparent bright-blue particles, while indigo, when viewed under the microscope, is more of a greenish blue. The detached particles of coloring matter often rise to the surface of the liquid, when the leaves are shaken in hot water, and for microscopical examination may be floated upon a glass slide. The color of Prussian blue is discharged by treatment with sodium hydroxide, while that of indigo is not. Prussian blue, if present, may be chemically detected in the sediment as above obtained, by dissolving in hot alkali, acidifying with hydrochloric acid, and then adding a drop of ferric chloride. A blue precipitate is indicative of the ferric ferrocyanide.

Such minerals as gypsum and soapstone are readily separated as a sediment by shaking the leaves in water, and the sediment is examined by the appropriate qualitative methods for these substances.

Spent or Exhausted Leaves.—These consist of leaves of tea that have been previously steeped or infused, and afterwards rerolled and dried. Such leaves are sometimes mixed with tea as an adulterant. Any considerable admixture of spent leaves is evident, both by the extremely low ash, and the abnormally small proportion of water-soluble ash in the sample. It is rare that the total ash of genuine tea is under 5%, while the soluble ash is seldom less than 3%.

The ash of spent tea leaves sometimes runs as low as 2.5%, of which generally not more than 0.3 to 0.8 per cent is soluble. Spent leaves are also naturally low in tannin and in extract.

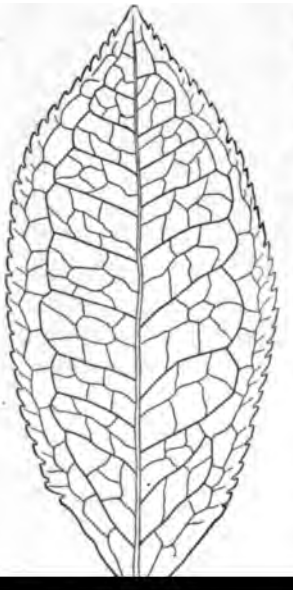
If the extract is much below 32%, spent leaves may be suspected. Allen has suggested the following formula for determining the percentage of spent leaves, E, in a sample of tea, R being the percentage of extract:

$$E = \frac{(32 - R)100}{30}.$$

The use of spent or exhausted leaves as an adulterant is very rare at present, though formerly of common occurrence.

Foreign Leaves as a Substitute for Tea.—This sophistication is not common, but the detection of leaves other than tea is readily accomplished by a careful examination of the shape and character of the leaves. For this purpose the dried leaves are opened out by soaking a short time in hot water, after which they are spread upon a glass plate, and examined by the aid of a magnifying-glass.

The genuine tea leaf (Fig. 67) is very characteristic, and is readily distinguished from other leaves. It is oval or lanceolate, 5 to 8 cm. long and 2 to 3 cm. wide. It is short-stemmed, somewhat thick and fleshy, attenuated at the bottom and usually pointed at the top. At a certain height from the base, from a third to a quarter up, the smooth or wavy border becomes peculiarly, though not deeply, serrated in a regular manner, the serrations, which are hook-shaped, continuing to the tip of the leaf. Mature leaves always show these serrations, but they are somewhat obscure in young leaf buds. The latter, however, are rarely found in this country. The veins extend outward from the central rib nearly parallel to each other, but before reaching the border, each bends upward to form a loop with the one above.



Foreign leaves, said to be used as adulterants are those of the willow, poplar, elder,

The ash of such "tea" has been found as high as 50%. Such imitations are now almost unknown. Make-weight substances, such as brick-dust, iron salts, metallic iron, sand, etc., have been found in tea. If present, they are to be found in the sediment, obtained on shaking out the tea in water.

Added Astringents.—Catechu is sometimes said to be added to tea to give it increased astringency, especially to such tea as has been adulterated by the addition of exhausted tea. Hagar's method for detecting catechu is as follows:

A hot-water extract of the tea (1 to 100) is boiled with an excess of litharge and filtered. To a part of the filtrate, which should be perfectly clear, nitrate of silver is added. If catechu be present, a yellow flocculent precipitate, rapidly becoming dark-colored, is formed. Pure tea treated in like manner gives a gray precipitate.

Spencer* adds, instead of silver nitrate, a drop of ferric chloride to the clear filtrate. With catechu a green precipitate is formed.

As a matter of fact the worst forms of tea adulteration, such as the actual substitution of foreign leaves, once so commonly practiced, are now extremely rare in this country and have been for some years, by reason of the careful system of government inspection in force at the various ports of entry. The greater portion of the tea on our market to-day is genuine, but fraud is practiced to a considerable extent by the substitution of inferior grades for those of good quality. This form of deception is in many cases beyond the power of the analyst to detect, and properly comes within the realm of the professional tea-taster.

Tea Tablets.—Finely ground tea of varying quality is sometimes pressed into tablets, to be used by travelers and campers for preparing a beverage, by simply dissolving in hot water.

The composition of one of these preparations sold under the name of Samovar Tea Tablets, analyzed by the Mass. State Board of Health, is as follows:

Water	8.7
Theine	2.25
Extract	54.4
Ash	5.4
Soluble ash	2.8
Insoluble ash	2.6

* U. S. Dept. of Agric., Div. of Chem., Bul. 13, p. 885.

Microscopical Structure of Tea.—The powdered tea may be examined directly in water-mount. Schimper recommends treating the powdered tea with chloral hydrate or potash lye, to render it more transparent.

By far the most characteristic element is the peculiarly shaped sclerenchyma, or stone cell, *st*, Fig. 68, entirely unlike anything to be found in other leaves. These cells are very irregular in form, being sometimes star-shaped, sometimes branched, almost always with deeply wrinkled sides,

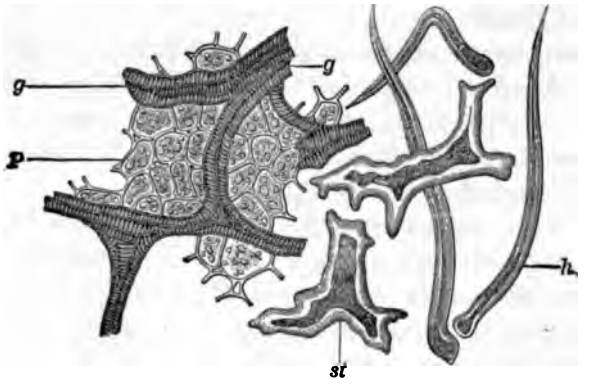


FIG. 68.—Powdered Tea under the Microscope. ×160. *g*, end of leaf nerve; *p*, chlorophyll parenchyma; *st*, stone cells; *h*, hairs. The tissues were warmed in potash to render transparent. (After Moeller.)

and often with sharp points. In most foreign leaves such sclerenchyma cells are lacking, but they are abundant in all genuine tea leaves, excepting rarely in the very young leaves, where they are sometimes not fully developed. They are especially numerous in the main vein and in the stem. They may be seen to best advantage in a section of the stem, or midrib,

COFFEE.

Nature of Coffee.—Coffee is the seed of the *Coffea arabica*, a tree which, when under cultivation, is not allowed to exceed twelve feet in height, but when wild sometimes reaches a height of twenty feet. It is indigenous in Southern Abyssinia, and was cultivated in Arabia in the sixteenth century, and in the East Indies in the seventeenth, afterward being introduced into the West Indies and South America. The coffee-beans are usually inclosed in pairs in the berry, being plano-convex with their flat sides together.

When the ripe fruit is gathered, it is first dried and then freed from the hulls, usually by machinery, or, in the West Indies, the green berries are "pulped" or "hulled" under water by a peculiar macerating machine. The raw beans are roasted, and afterwards ground for preparing the infusion.

Brazil furnishes more than half the world's supply of coffee, and nearly 75% of that consumed in the United States.

Composition of Coffee.—Most of the coffee in the retail market is roasted, being sold either in the whole bean or ground. Comparatively little raw coffee is sold at retail.

The constituents of raw coffee, besides water, are, in the order of their comparative amounts, cellulose or crude fiber, fat, protein, caffetannic acid, sugar, caffein, gum, dextrin, and ash. The effect of roasting coffee, besides driving off most of the water, is to caramelize a large part of the sugar, to make the bean less tough and more brittle, and, most important of all, to develop an empyreumatic, oily substance, known as *caffeol* ($C_8H_{10}O_2$), to which the characteristic flavor and aroma of coffee are largely due. Caffeol may be obtained by distilling an infusion of roasted coffee, and extracting the distillate with ether. It is a brown oil, almost insoluble in water.

According to Génin, there are in raw coffee small amounts of two essential oils, one soluble in water, the other insoluble. During the roasting, these are partially transformed into the substance caffeol.

The fat in coffee forms a considerable constituent, amounting in some cases to 15%.

Caffetannic Acid ($C_{15}H_{18}O_8$) is, when pure, a colorless, crystalline compound. It exists in coffee either as a salt of calcium or magnesium, or, according to Payen, as a caffetannate of potassium and caffeine.

The following summary of analyses of coffee of various kinds made by König show in general its composition:

	Raw Coffee.		Roasted Coffee.	
	Minimum.	Maximum.	Minimum.	Maximum.
Water	8.0	12.0	0.4	4.0
Caffeine	0.8	1.8	0.8	1.8
Fat	11.4	14.2	10.5	16.5
Reducing sugar	5.8	7.8	0.0	1.1
Cellulose	16.6	42.3	26.3	51.0
Total nitrogen	1.1	2.2	1.3	2.7
Ash	3.5	4.0	4.0	5.0

The change in composition that takes place in roasting coffee is well shown by the following figures, which give the mean of analyses by König of four samples of coffee before and after roasting:

	Raw Coffee.	Roasted Coffee.
Water	11.23	1.15
Caffeine	1.21	1.24
Fat	12.27	14.48
Sugar	8.55	0.66
Cellulose	18.17	10.89
Nitrogenous substances	12.07	13.98
Other non-nitrogenous matter	32.58	45.09
Ash	3.92	4.75

COMPOSITION OF THE ASH OF COFFEE.*

Constituents.	Mocha.	Maracaibo.	Java.	Rio.
Sand	1.44	0.72	0.74	1.34

	Mocha Coffee.		East Indian Coffee.	
	Raw.	Roasted.	Raw.	Roasted.
Moisture	8.98	0.63	9.64	1.13
Caffeine	1.08	.82	1.11	1.05
Saccharine matter	9.55	.43	8.90	.41
Caffeic acid	8.46	4.74	9.58	4.52
Alcoholic extract	6.90	14.14	4.31	12.67
Fat and oil	12.60	13.59	11.81	13.41
Legumin and albumin	9.87	11.23	11.23	13.13
Dextrin	.87	1.24	.84	1.38
Cellulose and insoluble coloring matter	37.95	48.62	38.60	47.42
Ash	3.74	4.56	3.98	4.88

Methods of Analysis.—*Moisture and ash*, both soluble and insoluble, as well as *caffeine* and *total nitrogen*, are estimated as in the case of tea (q.v.).

Fat.—Two grams of the finely powdered, dried sample are extracted in a Soxhlet apparatus with dry, alcohol-free ether or petroleum ether, till the soluble portion is removed, after which the ether is distilled off from the residue, and the latter is weighed. This residue consists of both fat and caffeine. The latter is estimated in the residue and deducted from the total weight.

Crude Fiber.—This is determined by subjecting the residue from the ether extract to the regular process for crude fiber (p. 218).

Caffetannic Acid.—*Krug's Method.**—2 grams of the coffee are digested for thirty-six hours with 10 cc. of water, after which 25 cc. of 90% alcohol are added, and the digestion continued for twenty-four hours more. The liquid is then filtered, and the residue washed with 90% alcohol on the filter.

The filtrate, which contains tannin, caffeine, fat, etc., is heated to boiling, and a boiling concentrated solution of acetate of lead is added, which precipitates out a caffetannate of lead, $Pb_3(C_{15}H_{15}O_{82})$, containing 49% of lead. When this has become flocculent, it is separated by filtration, and washed on the filter with 90% alcohol, till the washings show no lead with ammonium sulphide, and afterwards with ether, till free from fat. It is dried at 100° and weighed.

$$\text{Weight of caffetannic acid} = \frac{\text{Wt. of precipitate} \times 652}{1263.63}.$$

* U. S. Dept. of Agric., Div. of Chem., Bul. 13, p. 908.

ADULTERATION OF COFFEE.

The standard for pure coffee proposed by the A. O. A. C. is as follows: Standard roasted coffee should contain not less than 10% of fat, nor more than 15% of carbohydrates; ash should not be less than 3%, of which at least 75% is soluble in water.

Whole coffee is, as a rule, rarely adulterated at the present day, though the substitution of cheaper or inferior brands for the choicer varieties is a common practice. Formerly, artificial coffee-beans containing no coffee whatever, but cleverly molded to imitate the original, were occasionally to be found, mixed with genuine, whole coffee.*

"Coffee pellets" are occasionally sold in bulk to dealers as an adulterant of whole coffee. These do not closely resemble the real berries in appearance, but are approximately of the same size, and are not apparent to the purchaser when the whole coffee is ground at the time of purchase. A sample of these "pellets" examined recently was found to consist of roasted wheat mash, colored with red ocher.

Adulterants to be met with in ground coffee are legion. In Massachusetts the following have been found: Roasted peas, beans, wheat, rye, oats, chicory, brown bread, pilot bread, charcoal, red slate, bark, and dried pellets, the latter consisting of ground peas, pea hulls, and cereals, held together with molasses.

Methods of Detecting Adulterants.—These methods are, as a rule, physical rather than chemical. A rough test of the genuineness of ground coffee consists in shaking some of the sample in cold water. Pure coffee, under these conditions, usually floats on the surface, while the ordinary adulterants, such as cereals, chicory, mineral ingredients, etc., sink,



A careful examination of the coarsely crushed grains of a ground sample with the naked eye will often serve to detect, and in some cases identify, certain adulterants, such as chicory and ground peas or beans. A magnifying-glass will aid in such an examination, and the observer can often separate the various ingredients of a coffee mixture, first spreading a small portion of the sample on a sheet of white paper. The chicory grains are apparent from their dark and somewhat gummy appearance, and can usually be recognized by crushing them between the teeth. Their soft consistency and bitter taste are very distinctive. The dull surface of the outside of the crushed coffee grains is in marked contrast to the polished appearance of the surface of the broken peas or beans, often to be found as adulterants, while fragments of broken cereal grains are readily distinguished from coffee with a low-power magnifier, though perhaps not easily identified by the eye alone.

Determination of Added Starch.—Starch is determined in the finely powdered sample as directed on page 223.

Microscopical Examination of Coffee.—By far the best and most trustworthy means for the analyst to employ in the examination of coffee for its adulterants is furnished by the microscope. For the purposes of microscopical examination, a small portion of the sample is pulverized in a mortar to a degree fine enough to allow the cover-glass to lie flat on the wetted sample, yet not so fine that it ceases to feel granular when rubbed between the fingers. If powdered too fine, some of the elements are too fragmentary to be readily recognizable. The writer finds it sufficient to examine this powder in water-mount without further treatment.

Schimper recommends maceration for twenty-four hours with ammonia, in order to render the tissues more transparent, using this reagent also as a mountant. In general the interior of the coffee tissue or endosperm consists of irregular polygonal parenchyma cells, (1), Fig. 69, sometimes with 4 and sometimes with 5 sides.

To a large extent the cells are in fragments, but are easily recognizable. They contain brilliant, colorless, spherical oil drops, and also albumin. The cell walls are frequently sinuous with irregular projections.

The seed coat is also very characteristic, showing in the powder as occasional delicate silver-like patches, with peculiar, spindle-shaped, thick-sided cells, some of which are loosened from the tissue.

Plates XIV and XV illustrate photomicrographs of pure and adulterated coffee. Fig. 174 shows genuine coffee, with its loose mesh of irreg-

ularly pentagonal cells, thick-walled, and inclosing oil drops with amorphous material. It is not to be expected that every pulverized sample of genuine coffee, mounted as above, will show in every microscopic field the even, continuous structure that Fig. 174 illustrates, but careful examination will

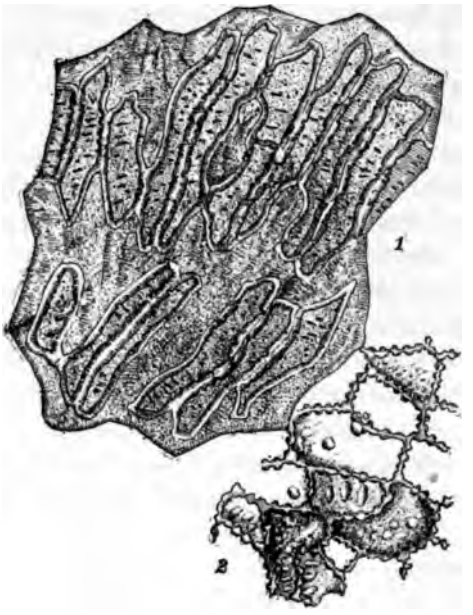


FIG. 69.—Powdered Coffee under the Microscope. ×125. (After Moeller.) 1, seed coat (surface). 2, endosperm parenchyma.

by means of which the latter may be sometimes distinguished from allied roots, as the dandelion, etc.

Fig. 178, Pl. XV, is a photomicrograph of an adulterated sample of coffee, showing in this particular field chicory alone. It is a mass of con-

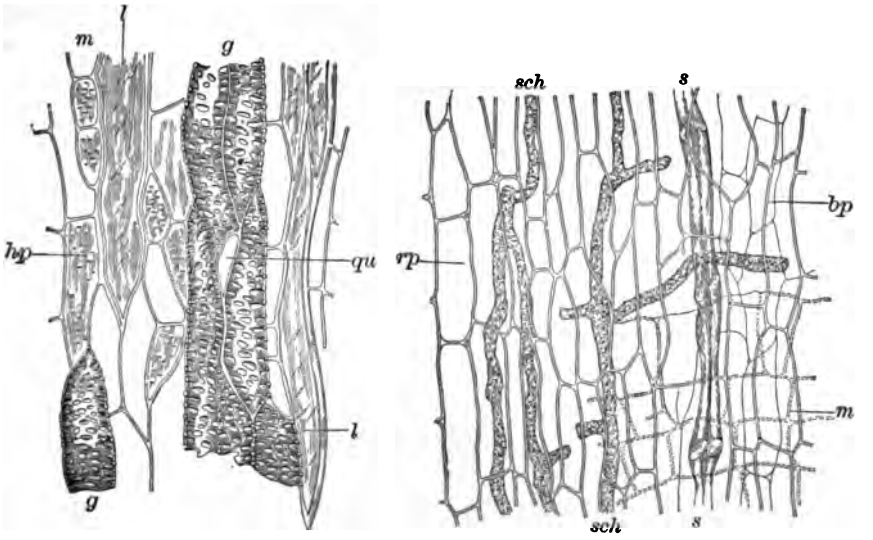


FIG. 70.—Chicory Root in Tangential and Radial Sections. ×160. *g*, reticulated ducts with perforations *qu*; *hp*, wood parenchyma; *l*, wood fibers; *rp*, bark parenchyma; *sch*, milk ducts; *bp*, bast parenchyma; *m*, medullary rays. (After Moeller.)

fused cellular tissue, traversed by two broad bands of the sieve ducts, with their striking, transverse, dotted markings.

Fig. 177, Pl. XV, shows a sample of coffee adulterated with roasted peas and pea hulls. No genuine coffee appears in this field. The chief masses in the center are characteristic aggregations of the round starch granules of the roasted pea. The rectangular billets, like bunches of matches, are pea hulls, or the cells composing the skin of the pea.

Fig. 164, Pl. XI, and Fig. 154, Pl. IX, show the close resemblance between the starches of the pea and bean, both of which are commonly used in coffee.

The "billets," or palisade structure of the hulls of these legumes also bear a close resemblance. Moeller has shown that the average length of the pea "billets" is 100 micromillimeters, while those of the bean hulls average 45.

The effect of roasting on starches used as adulterants of coffee is to twist and distort the granules, in some cases destroying largely the even

structure of the raw starch. Starch granules of wheat, barley, and rye, for example, are almost perfect circular disks in the case of the raw starch, while in the roasted product, the granules are twisted and distorted, sometimes almost forming the letter "S."

Ground pilot crackers and stale breads show this distorted structure.

Use of Chicory in Coffee.— Chicory is a perennial herb (*Cicorium intybus*) of the same family (*Compositæ*) as the dandelion. The roasted and pulverized chicory root is so much used in ground coffee to impart a peculiar flavor thereto, that by many it is considered as not strictly an adulterant. The taste imparted to coffee by a small admixture of pure chicory is to some desirable, but if its unrestricted use is sanctioned in this manner, the door would soon be opened to a more unlimited form of adulteration, wherein the chicory might predominate. It is, therefore, best to regard chicory as an adulterant, and to require the package containing a mixture of coffee and chicory, if sold legally, to have plainly printed thereon the percentage of chicory in the mixture.

Chicory, when roasted, consists of gum, partly caramelized sugar, and insoluble vegetable tissue.

Villiers and Collin * give the following analyses of two samples of chicory:

		In Large Granules.	In Powder.
Soluble in water:	Water (loss at 100° to 103°)	16.28	16.96
	Weight of total matter soluble in water	57.96	56.90
	Reducing sugar	26.12	23.79
	Dextrin, gum, inulin	9.63	9.31
	Albuminoids	3.23	3.66
	Mineral matter	2.58	2.55
	Coloring matter	16.40	17.59
	Albuminoids		

of the latter can be calculated from the specific gravity of a 10% decoction, using conveniently the method of McGill.* A quantity of the pulverized sample, corresponding to 10 grams of the dry substance, is weighed in a counterbalanced flask, and water added till the weight of the contents is 110 grams. Fit the flask with a reflux condenser, and after so regulating the heat that boiling begins in ten to fifteen minutes, continue the boiling for an hour. Remove the flame, and after fifteen minutes pass through a dry filter, cool, and determine the specific gravity at 15°. McGill found the average specific gravity of a 10% decoction as above carried out to be, in the case of pure coffee, 1.00986 and in the case of chicory 1.02821, the difference being 0.01835.

The specific gravity of the 10% decoction of the suspected sample at 15° being d, the per cent of chicory, c, can be calculated roughly by the formula

$$c = 100 - \frac{(1.02821 - d)100}{0.01835}.$$

This method is of course inapplicable when other substances than chicory are present.

Date Stones, roasted and ground, have been used to some extent as a coffee adulterant. Fig. 71 shows the structural features of date stones

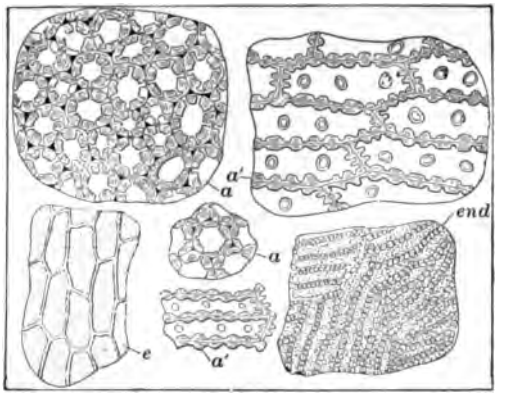


FIG. 71.—Powdered Date Stones under the Microscope. *end*, endocarp; *e*, episperm; *a*, albumen in cross-section; *a'*, albumen in longitudinal section. (After Villiers and Collin.)

under the microscope. *End* represents a fragment of endocarp with its elongated, thick-walled cells, peculiarly arranged as shown, adjacent cells

* Trans. Royal Soc. of Canada, 1887.

often lying with axes at right angles to each other. The more evenly formed episperm cells, *e*, are thin-walled and of a brown color. The albumen, *a*, is made up of very thick-walled, somewhat regularly arranged cells, indented from within with deep channels. Date stones are readily distinguished from coffee by these features.

Coloring Coffee Beans.—The practice of treating raw coffee beans in a manner somewhat analogous to the facing of tea leaves has been sometimes practiced, with a view to giving to cheaper or inferior grades the appearance of high-priced coffee. For this purpose various pigments have been employed, such as yellow ocher, chrome yellow, burnt umber venetian red, Scheele's green, iron oxide, turmeric, indigo, Prussian blue, etc., the coffee beans being first moistened with water containing a little gum, and shaken with the pigment. As a rule such pigments, especially when inorganic, are best sought for either in the ash, or in the sediment obtained by shaking the coffee beans in cold water, using the ordinary qualitative chemical methods. Organic coloring matters can be best extracted with alcohol. Prussian blue and indigo are tested for as in the case of tea leaves (p. 285).

Glazing.—This is a more recent form of treatment of the whole bean, which consists in coating the beans by dipping in egg or sugar, or a mixture of the two, sometimes using various gums. Such glazing is alleged to improve the keeping qualities of the coffee, as well as to aid in clarifying the infusion, and if this is the sole purpose, the practice cannot be condemned as a form of adulteration. If, however, it is done to give inferior varieties of coffee a better appearance, in order to deceive the consumer, it clearly constitutes adulteration within the meaning of the law.

Coffee Substitutes.—A large number of preparations sold as "coffee

ing them. In coffee substitutes, coffee itself should properly be considered in the light of an adulterant.

COCOA AND COCOA PRODUCTS.

Nature of Cocoa.—Cocoa and the various chocolate and cocoa preparations are made from the bean of the tree *Theobroma cacao*, of the family of *Byttneriaceæ*. This tree averages 13 feet in height, and its main trunk is from 5 to 8 inches in diameter. It is a native of the American tropics, being especially abundant and growing under best conditions in Mexico, Central America, Brazil, and the West Indies.

The cocoa beans of commerce are derived chiefly from Ariba, Bahia, Caracas, Cayenne, Ceylon, Guatemala, Haiti, Java, Machala, Maracaibo, St. Domingo, Surinam, and Trinidad. Besides these, the Seychelles and Martinique furnish a small amount.

The plant seeds, or beans, grow in pods, varying in length from 23 to 30 cm., and are from 10 to 15 cm. in diameter. The beans, which are about the size of almonds, are closely packed together in the pod. Their color when fresh is white, but they turn brown on drying.

The gathered pods are first cut open, and the seeds removed to undergo the process of "sweating" or fermenting, which is carried out either in boxes or in holes made in the ground. This process requires great care and attention, as upon it depends largely the flavor of the seed. The sweating operation usually takes two days, after which the seeds are dried in the sun till they assume their characteristic warm red color, and in this form are shipped into our markets.

For the production of the various preparations of chocolates and cocoa, the beans are cleaned and carefully roasted, during which process the flavor is more carefully developed, and the thin, paper-like shell which surrounds the seed is loosened, and is very readily removed. The roasted seeds are crushed, and the shells, which are separated by winnowing, form a low-priced product, from which an infusion may be made, having a taste and flavor much resembling chocolate.

The crushed fragments of the kernel or seed proper are called *cocoa nibs*, and for the preparation of chocolate they are finely ground into a paste and run into molds, either directly, or after being mixed with flavoring extract, and, in some cases, sugar and spices, according to whether plain or sweet chocolate is the end product.

For making cocoa, however, a portion of the oil or fat known as the

cocoa butter is first removed, by subjecting the ground seed fragments to hydraulic pressure, usually between heated plates, after which the pressed mass is reduced to a very fine powder, either directly, or by treatment with ammonia or alkalies, to render the product more soluble. It is held that the large amount of fat contained in the cocoa seeds (varying from 40 to 54 per cent) is difficult of digestion to many, such as invalids and children, and hence the desirability of removing part of the fat.

Composition of Cocoa.—The chief constituents of raw cocoa, named in the order of their relative amount, are fat, proteids, starch, water, crude fiber, ash, theobromine, gum, and tannin. During the roasting there is reason to believe a volatile substance is developed much in the nature of an essential oil, which gives to the product its peculiar flavor, and is somewhat analogous to the caffeol of coffee.

Tannin, the astringent principle of cocoa, exists as such in the raw bean, but rapidly becomes oxidized to form *cocoa red*, to which the color of cocoa is due.

Weigmann gives the following results of analyses of cocoa nibs and shells:

COMPOSITION OF COCOA NIBS.

Commercial Varieties.	Moisture.	Nitrogenous Substances, including Theobromine.	Theobromine.	Fat.	Starch.	Other Carbohydrates.	Cellulose.	Ash.	Sand.
Caracas.	7.77	14.13	1.48	45.54	19.40	.....	6.19	4.91	2.06
Trinidad.	7.87	14.06	1.31	44.62	25.30	.....	4.55	3.48	0.10
Surinam	7.53	13.69	1.66	44.74	26.45	.....	4.30	3.16	0.13
Port au Prince.	7.77	14.56	.....	46.35	5.97	15.53	5.19	4.15	1.48
Machata	8.17	14.06	.....	45.93	5.69	17.50	4.36	4.09	0.22

The following are the summarized results of the analyses of seventeen varieties of cocoa seeds and shells, made by Winton, Silverman, and Bailey.*

	Roasted Cocoa Nibs.					
	Air-dry Material.			Water- and Fat-free Material.		
	Maximum.	Minimum.	Mean.	Maximum.	Minimum.	Mean.
Water	3.18	2.29	2.72			
Total ash	4.15	2.61	3.32	8.81	5.76	7.04
Water-soluble ash	1.86	0.73	1.16	3.96	1.60	2.46
Ash insoluble in acid	0.07	0.00	0.02	0.14	0.00	0.05
Alkalinity of ash	3.35	1.50	2.51	7.12	3.20	5.32
Theobromine	1.32	0.82	1.04	2.92	1.66	2.21
Caffeine	0.73	0.14	0.40	1.55	0.31	0.86
Other nitrogenous substances	13.06	11.00	12.12	28.05	23.37	25.09
Crude fiber	3.20	2.21	2.64	6.56	4.70	5.61
Crude starch (acid conversion)	12.37	9.30	11.16	25.68	19.80	23.66
Pure starch (diastase conversion)	8.99	6.49	8.07	18.61	13.82	17.10
Other nitrogen-free substances	21.07	17.69	19.57	44.08	38.78	41.49
Fat	52.25	48.11	50.12			
Total nitrogen	2.54	2.20	2.38	5.41	4.74	5.05
Constants of fat (ether extract):						
Melting-point, degrees C.	35.0	32.3	33.3			
Zeiss refractometer reading at 40° C.	48.00	46.00	47.23			
Refractive index at 40° C.	1.4579	1.4565	1.4573			
Iodine number	37.89	33.74	34.97			
Per cent of nibs in whole bean	92.90	86.12	88.46			
" " " shells " " "	13.88	8.83	11.54			

	Roasted Cocoa Shells.					
	Air-dry Material.			Water- and Fat-free Material.		
	Maximum.	Minimum.	Mean.	Maximum.	Minimum.	Mean.
Water	6.57	3.71	4.87			
Total ash	20.72	7.14	10.48	21.97	5.63	11.33
Water-soluble ash	5.67	2.02	3.67	6.11	2.16	3.97
Ash insoluble in acid	11.18	0.05	2.51	11.86	0.05	2.70
Alkalinity of ash	5.92	5.02	5.52	6.47	5.32	5.97
Theobromine	0.90	0.20	0.49	0.97	0.22	0.52
Caffeine	0.28	0.04	0.16	0.31	0.04	0.17
Other nitrogenous substances	18.06	10.69	14.54	19.40	11.34	15.70
Crude fiber	19.21	12.93	15.63	20.72	13.71	18.01
Crude starch (acid conversion)	13.89	9.87	11.62	15.42	10.47	12.59
Pure starch (diastase conversion)	5.16	3.36	4.14	5.59	3.65	4.47
Other nitrogen-free substances	51.86	43.71	46.40	55.84	47.04	50.08
Fat	5.23	1.66	2.77			
Total nitrogen	3.17	1.74	2.34	3.41	1.87	2.54

* An. Rep. Conn. Agric. Exp. Sta., 1902, p. 270.

According to Bell* the ash of cocoa nibs has the following composition:

	Per Cent.
Sodium chloride	0.57
Soda	0.57
Potash	27.64
Magnesia	19.81
Lime	4.53
Alumina	0.08
Ferric oxide	0.15
Carbonic acid	2.92
Sulphuric acid	4.53
Phosphoric acid	39.20
	100.00

Theobromine ($C_7H_8N_4O_2$), the chief alkaloid of cocoa, when pure, forms a white, crystalline powder, having a bitter taste. It is slightly soluble in water and alcohol, very slightly soluble in ether, insoluble in petroleum ether, but readily soluble in chloroform. It sublimes at 290° to 295° C. It is a weak base, and much resembles caffeine. A small amount of caffeine has also been found in cocoa, but in most analyses is reckoned in with the theobromine.

The Nitrogenous Substances of Cocoa, aside from the alkaloids, have been little studied. Stutzer has, however, separated them roughly as in the following analyses of four samples, of which A was manufactured without chemicals, B with potash, and C and D with ammonia:

	A.	B.	C.	D.
Total nitrogen	3.68	3.30	3.05	3.57

Ash.—The dish containing the above residue is brought to a dull-red heat over a low Bunsen flame, and kept at this temperature till a white ash is obtained, after which it is cooled in a desiccator and weighed. The ash is then boiled with water, brought upon a filter, washed, reignited, and the insoluble portion weighed.

If the ash is to be examined for mineral adulterants, such as red ocher, etc., a much larger amount of the original substance should be taken.

Fat.—Five grams of the sample, after drying, are extracted with petroleum ether in a Soxhlet apparatus, and the residue, separated by evaporation from the solvent, is dried at 100° C. Dry, alcohol-free ether is sometimes used as a solvent, but does not give accurate results, since theobromine is slightly soluble in boiling ether (about 1 part in 600).

Fat thus extracted from chocolate should be examined for purity (see p. 304), since from the high price of cocoa butter, the fat is said to be sometimes expressed and replaced in part by cottonseed stearin.

Theobromine.—Total alkaloid, reckoned as theobromine, may be determined approximately by the same method as given for caffeine (p. 281). The separation of the small amount of caffeine present from the theobromine, and its determination, are rarely considered necessary, so similar are the two alkaloids in their nature and effects.

Schmidt and Pressler* employ the following method: Ten grams of the sample are freed from fat by extracting with petroleum ether in a Soxhlet apparatus. The residue is mixed with an equal quantity of slaked lime, and the mixture repeatedly boiled with 80% alcohol. The residue left on evaporating off the alcohol is best recrystallized from the same solvent, and should be in the form of a white, crystalline powder. It is dried at 100° and weighed. Caffeine, if present, is included with the theobromine.

Methods for the Separation of Caffeine and Theobromine.—The mixed alkaloids are best extracted with cold benzol, which dissolves the caffeine, but in which theobromine is practically insoluble.

Maupy's Method for Estimation of Theobromine.†—Extract the fat from 5 grams of cocoa, or from 10 grams of the chocolate with petroleum ether. Triturate the residue in the case of the cocoa with 2 cc. of water, or, in the case of the chocolate, with 4 grams 70% alcohol. Boil for an hour with a mixture of 15 grams phenol and 85 grams chloroform under a reflux condenser. Cool, filter off the extract, and shake the residue

* Ann. der Chem., 217, 288.

† Jour. Pharm. Chem., 1897, V, 329–332; Abs. Analyst, XXII, 191.

twice with 15 grams of chloroform, allowing it to macerate half an hour each time, and adding the filtered chloroform to the original phenol-chloroform extract. Evaporate off the chloroform, add 40 grams of ether to the residue, and let stand for six hours. This precipitates the theobromine, while the caffeine, coloring substances, and any traces of fat, remain in solution. Collect the precipitate on a tared filter, wash with a little ether, dry, and weigh.

Albuminoids.—The total nitrogen is determined by the Gunning method (p. 61), but if the nitrogen be multiplied by 6.25, the result, counted as albuminoids, is too high by reason of the theobromine. Best results are obtained by first removing the alkaloids by treatment with chloroform, and determining the nitrogen in the residue by the regular Gunning method.

Starch.—Wash 4 grams of the sample on a filter-paper with ether till free from fat. Then wash with cold water on the same filter till free from sugar. Subject the residue to the diastase treatment for starch, p. 223.

Crude Fiber.—Two grams of the sample are freed from fat and the regular method of the A. O. A. C. (p. 218) employed.

Cocoa Butter.—The fat of the cocoa, known as cocoa or cacao butter, is much used in pharmacy as a basis for ointments and to some extent in soap-making, so that there is a temptation to remove the fat on the grounds of its commercial value, as well as for the alleged reason of rendering the cocoa more digestible. Cocoa butter is sometimes adulterated with paraffin wax and cheaper fats, such as cocoanut oil, cottonseed, stearin, and tallow. Tests for its purity consist in determining the melting-point, that of pure cocoa butter being 28° to 33° C., the iodine absorption value (which should lie between 32 and 41), the saponification value (about 192.5), the specific gravity, which at 15° is 0.9599–0.976, and the



and fat-free residue no higher percentage of either ash, fiber, or starch than is found in the sugar- and fat-free residue of plain chocolate.

Standard cocoa should contain percentages of ash, crude fiber, and starch corresponding to those of plain chocolate, after correcting for fat removed.

Standard sweet cocoa is cocoa mixed with sugar (sucrose) containing not more than 60% of sugar, and in the sugar- and fat-free residue no higher percentage of either ash, crude fiber, or starch than is found in the sugar- and fat-free residue of plain chocolate.

The removal of fat, or the addition of sugar beyond the above prescribed limits, or the addition of foreign fats, foreign starches, or other foreign substances, constitutes adulteration, unless plainly stated on the label.

The most common adulterants of cocoa are sugar and various starches, especially those of wheat, corn, and arrowroot. Starch is sometimes added for the alleged purpose of diluting the cocoa fat, instead of removing the latter by pressure, thus, it is claimed, rendering the cocoa more digestible and more nutritious. Unless its presence is announced on the label of the package, starch should be considered as an adulterant. Cocoa shells are also commonly employed as a substitute for, or an adulterant of, cocoa. Other foreign substances found in cocoa are sand and ground wood fiber of various kinds. Iron oxide is occasionally used as a coloring matter, especially in cheap varieties.

Such adulterants as the starches and cocoa shells are best detected by the microscope. The presence of any considerable admixture of sugar is made apparent by the taste. Mineral adulterants are sought for in the ash.

Addition of Alkali.—The amount of water-soluble matter in cocoa is very small (about 20 to 25 per cent), and in preparing the beverage, the desideratum aimed at is to produce as perfect an emulsion as possible. The legitimate means of accomplishing this is by pulverizing the cocoa very fine, so that particles remain in even suspension and form a smooth paste. Another means sometimes resorted to for producing a so-called "soluble cocoa" is to add alkali in its manufacture, the effect being to act upon a part of the fat, and produce a more perfect emulsion with less separation of oil particles. Such treatment with alkali is regarded with disfavor, even if not considered as a form of adulteration. Cocoa thus treated is generally darker in color than the pure article.

The use of alkali is usually rendered apparent by the abnormally high

ash, and by the increased alkalinity of the ash, the latter constant being expressed in terms of the number of cubic centimeters of decinormal acid necessary to neutralize the ash of 1 gram of the sample. In pure, untreated cocoa, the ash rarely exceeds 5.5%, and the alkalinity of the ash is generally not more than 3.75. In cocoa treated with alkali, the ash sometimes reaches 8.5%, with the alkalinity running as high as 6 or even 8.

Microscopical Structure of Cocoa.—Fig. 72 shows elements of the powdered cocoa bean, both of the shell and of the kernel. The powder of the latter should constitute pure cocoa, with occasional fragments only of the shell. The irregular lobes constituting the kernel are each inclosed in a membrane made up of angular cells, filled with granular matter. (4), (5), and (6) show elements of the powdered cotyledons, or seed kernels. The polygonal tissue of the cotyledon is shown in cross-section at (4). In the powder one finds also dark granular matter, bits of débris, and fragments, with masses of yellow, reddish-brown, and sometimes violet coloring matter, together with numerous starch granules and aleurone grains.

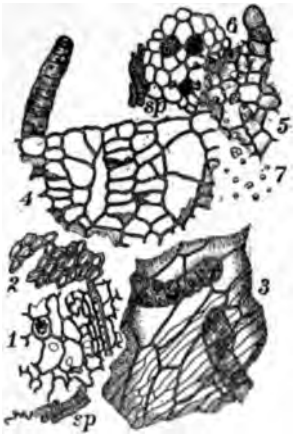


FIG. 72.—Powdered Cocoa under the Microscope. ×125. (After Moeller.) 1, cross-section through shell parenchyma; 2, thick-walled cells; 3, epidermis

The starch granules are nearly circular, with rather indistinct central nuclei, and range in size from 0.0024 to 0.0127 mm., averaging about 0.007 mm. They are more

vessel for twenty-four hours, before all the elements, of pure cocoa are rendered transparent. If after that time opaque masses are still found, these are due to foreign material.

Ammonia may be used instead of chloral with even better results, but this reagent requires longer treatment, soaking for several days or a week being sometimes necessary.

Fig. 185, Pl. XVII, shows the microscopical appearance of genuine powdered cocoa with its variously sized starch grains and the débris of the ground cotyledons. Fig. 186 shows cocoa adulterated with arrowroot.

Cocoa Shells.—The shell parenchyma with side view of the stone-cell layer *a* and frequent spiral ducts, all characteristic of the ground shell, are shown at 1, Fig. 72.

In plan view the thick-walled stone-cell layer is shown at 2, and the spongy, outer seed-skin tissue, composed of two layers, with elongated cells running crosswise to each other in striated fashion, and with the

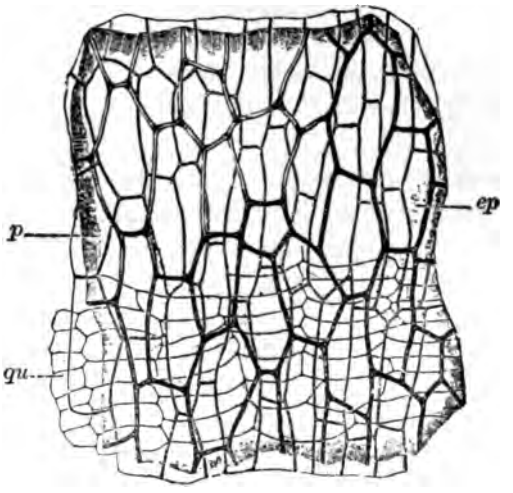


FIG. 73.—Cocoa Shell in Surface Section. ×160. *ep*, epicarp; *p*, parenchyma of the fruit; *qu*, layer of transverse cells. (After Moeller.)

underlying hairs or so-called "Mitscherlich bodies," is shown at 3. The presence of an abnormally large number of yellow and brown fragments in the water-mounted cocoa specimen, even under small magnification, arouses suspicion of the presence of shells, the most distinctive elements of which are the spongy tissue, the stone cells, and the abundant spiral ducts, the latter being scarce in pure cocoa powder.

Cocoa shells are indicated on chemical analysis by the abnormally high ash and crude fiber.

Added Starch.—This can only be approximately determined by a careful examination with the microscope. Long experience will enable the analyst to familiarize himself with the appearance and abundance of starch grains of various kinds in a series of fields, so that he can roughly estimate the amount of each starch present in the mixture, by careful comparison with mixtures of known percentage composition.

Added Sugar.—Any appreciable amount of added cane sugar is shown by the sweet taste. The amount of cane sugar may be determined by means of the polariscope, using preferably the double-dilution method of Wiley and Ewell, on account of the volume of the residue. For this purpose, extract separately with petroleum ether on filters quarter- and half-normal weights of the sample (6.512 and 13.024 grams), and make up to 50 and 100 cc. respectively, after clarification with subacetate of lead and alumina cream. Polarize in both cases in the 100-mm. tube, and multiply the readings by 4 for correction.

Subject each solution to inversion, and from the true direct and invert readings, obtained by Wiley and Ewell's formula, calculate the sucrose by Clerget's formula.

If a polariscope is not at hand, the sugar may be determined by extracting a weighed amount of the sample with cold water, and determining the reducing sugar, both before and after inversion, by either of the usual methods, the difference being the equivalent of the cane sugar.

An abnormally low ash is indicative of the addition of starch or sugar or both.



MACFARLANE, T. Coffee. Canada Inl. Rev. Dept. Buls. 3, 29, 31.
McGILL, A. Cocoa and Chocolate. Canada Inl. Rev. Dept. Bul. 72.
MICHAELIS, A. Der Kaffee als Genuss- und Heilmittel nach seinen botanischen, chemischen, dietetischen und medicinischen Eigenschaften.
PEARMAIN, T. H., and MOOR, C. G. On the Adulteration of Coffee. Analyst, 1895, 20, 176.
SMETHANE, A. Composition of Some Samples of Pure Coffee. Analyst, VII, 1882, page 73.
SPENCER, G. L. Tea, Coffee, and Cocoa Preparations. Div. of Chem. Bul. 13, Part VII, 1892.
TRILLICH, H. Die Kaffeesurrogate, ihre Zusammensetzung und Untersuchung. Munich, 1889.
WANKLYN, J. A. Tea, Coffee, and Cocoa. London, 1883.
WIGNER, G. W. Nitrogenous Constituents of Cocoa. Analyst, IV, 1879, page 8.
WINTON, A. L., SILVERMAN, M., and BAILEY, E. M. Cocoa. An. Rep. Conn. Exp. Sta., 1902, page 248.

Conn. Exp. Sta. Annual Reports, 1896 et seq.
Maine Exp. Sta. Bul. 65. Analysis of Coffee Substitutes.
Massachusetts State Board of Health Reports, 1882 et seq.
North Carolina Exp. Sta. Bul. 154. Adulteration of Coffee and Tea.
Penn. Dept. of Agric. An. Rep., 1897, page 178. Substitutes for Coffee.
" " " " " " 1898, pages 75 and 548. Coffee and its Adulterations.
" " " " " " " pages 90 and 652. Chocolate and Cocoa.

CHAPTER XI.

SPICES.

THESE aromatic vegetable substances are classed as condiments, and depend for their use on the pungency which they possess in giving flavor or relish to food. As such seasoning or zest-giving substances, they are of considerable importance dietetically, but from the fact that they are used in comparatively insignificant amount, the determination of their chemical composition or actual value as nutrients *per se* is of little importance to the food economist.

Spices are, however, of chief interest to the public analyst, because of all food materials they constitute from their nature a class more susceptible than others to fraudulent adulteration of the most skilled variety.

In many cases not only the megascopic appearance and taste of the skillfully adulterated article are made to counterfeit the genuine spice, but even the microscopical appearance is intended to deceive, since it is the microscope that is most useful in the detection of adulteration, and in many cases in the determination of the approximate amount of the adulterants.

Indeed it is very rare that the microscope will fail to detect the presence of any foreign substance in spice, and hence its use is indispensable in the study of this class of foods by the analyst. Chemical methods, as

under the discussion of the spice in question. For these determinations the spices should be powdered fine enough to pass through a 60-mesh sieve.

Determination of Moisture.—*Richardson's Method.**—Two grams of the sample are weighed in a tared platinum dish and dried in an air-oven at 110° to a constant weight, which generally requires about twelve hours. The loss in weight includes the moisture and the volatile oil. The latter is determined from the ether extract, as described on page 312, and deducted from the total loss to obtain the moisture.

McGill † determines the moisture by exposure of a weighed portion of the sample *in vacuo* over perfectly colorless sulphuric acid. The spice gives up its moisture before the volatile oil comes off, and any appreciable amount of the volatile oil, when absorbed by the acid, causes the latter to be discolored, so that by carefully observing the beginning of the discoloration, and removing the sample, the loss due to moisture may be obtained by weighing at the proper stage. The abstraction of the moisture in this manner requires about twenty-four hours.

Determination of Ash.—The residue from the determination of moisture is burnt to a white ash in the original platinum dish, either directly over a low flame, or preferably in a muffle. If it is impossible to obtain a white ash, the charred mass is exhausted with water, a little ammonium carbonate solution is added to the aqueous extract, and the latter is evaporated to dryness, the insoluble residue, which has been collected on a filter and burnt, is added, and the whole is incinerated to as near a white ash as possible.

The Water-soluble Ash ‡ is found by boiling the total ash as above obtained with 50 cc. of water, and filtering through asbestos in a tared Gooch crucible, the insoluble residue being washed with hot water, dried, ignited, and weighed.

The insoluble ash, subtracted from the total, leaves the water-soluble ash. When sand is present, its amount is assumed to be the percentage of ash insoluble in hydrochloric acid. The ash from 2 grams of the substance, obtained as above described, is boiled with 25 cc. of 10% hydrochloric acid (specific gravity 1.050) for five minutes, the insoluble residue is collected on a tared Gooch crucible, thoroughly washed with hot water, and finally dried and weighed.

---

* U. S. Dept. of Agric., Div. of Chem., Bul. 13, pt. 2, p. 165.

† Canada Dept. of Inland Rev. Bul. 73, p. 9.

‡ Winton, U. S. Dept. of Agric., Bur. of Chem., Bul. 65, p. 55.

Lime is determined from the ash as directed on page 264, having first separated the iron and phosphates.

The *sulphuric acid* due to calcium sulphate (added as an adulterant) is determined by precipitation with barium chloride of a very weak hydrochloric acid solution of the ash, the separated barium sulphate being washed, dried, ignited, and weighed.

Ether Extract—*Total, Volatile, and Non-volatile.**—Two grams of the powdered substance are placed in a Schleicher and Schull cartridge, or wrapped in fat-free filter-paper, and are subjected to continuous extraction in a Soxhlet apparatus for twenty-four hours with anhydrous, alcohol-free ether.† The ether solution is then transferred to a tared evaporating-dish, and allowed to evaporate spontaneously at the temperature of the room. After the disappearance of the ether, the evaporating-dish is placed in a desiccator over concentrated sulphuric acid and left over night, or for at least twelve hours, after which it is weighed, the residue in the dish being regarded as the total ether extract.

The dish and its contents are then subjected to a heat of about 100° C. for several hours, taking a long time to bring the temperature up to that point so as to avoid oxidation of the oil. Finally heat at 110° C. till the weight is constant. The final residue is the non-volatile, and the loss in weight the volatile ether extract.

Alcohol Extract.—*Method of Winton, Ogden, and Mitchell.*‡—Two grams of the powdered sample are placed in a 100-cc. graduated flask, which is filled to the mark with 95% alcohol. The flask is stoppered and shaken at half-hour intervals during eight hours, after which it is allowed to stand for sixteen additional hours without shaking, and the contents poured upon a dry filter. Of the filtrate, 50 cc. are evaporated to dryness in a tared platinum dish on the water-bath, and heated at 110° C.



Determination of Starch.—In spices like white pepper, ginger, and nutmeg that normally contain a high content of starch and very little other copper-reducing matter, the direct acid conversion process of starch determination is satisfactory.

In spices normally free from starch, such as cloves, mustard, and cayenne, where a starch determination indicates the amount of a foreign starch present as an adulterant, it is safer to use the diastase process.

Four grams of the powdered sample are extracted on a filter-paper (fine enough to retain all starch particles) first with five successive portions of 10 cc. of ether, then with 150 cc. of 10% alcohol. Owing to difficulty of filtering in the case of cassia and cinnamon, Winton recommends that all washing in the determination of starch in these substances be omitted. The residue is washed from the filter-paper by means of a stream of water into a 500-cc. flask, if the direct acid conversion method is used, using 200 cc. of water; 20 cc. of hydrochloric acid (specific gravity 1.125) are added, and the method from this point on followed, as detailed on page 223.

If the starch is to be determined by the diastase method, wash the residue from the filter-paper into a beaker with 100 cc. of water, and proceed as on page 223.

Determine the dextrose in either case by the Defren method, or volumetrically, and convert dextrose to starch by the factor 0.9.

Determination of Crude Fiber. — Two grams of the substance are extracted with ordinary ether (or the residue left from the determination of the ether extract may be taken) and subjected to the regular method for determining crude fiber, by boiling successively with acid and alkali (page 218).

McGill recommends the use of the centrifuge in separating the crude fiber, after boiling with the alkaline solution.

Determination of Volatile Oil.—*Method of Girard and Dupré.**—The spice is mixed with water and subjected to distillation, receiving the distillate in a graduated cylinder. The volume occupied by the essential oil (which is immiscible with water) can be thus read off and its content roughly determined. If the volatile oil is slightly soluble in water, separate out the water layer, having first read the volume of the oil layer, and extract the aqueous solution with petroleum ether. Evaporate the petroleum ether extract to dryness at room temperature

* Analyse des Matieres Alimentaires, page 655.

in a tared dish, and add the volume due to the weight of the residue to the volume read off in the graduate.

Microscopical Examination of Powdered Spices. — As a rule few microscopical reagents are necessary in the routine examination of powdered spices for adulteration, unless a more careful study of the structure than is necessary to prove the presence of adulterants is desirable. The simple water-mounted specimen is usually sufficient to show the purity or otherwise of the sample. If in doubt as to the presence of starch in small quantities, iodine in potassium iodide should be applied to the specimen, well rubbed out under the cover-glass.

Very rarely it is helpful to treat the spice powder with ammonia or chloral hydrate, to render certain of the hard opaque fragments more transparent. When this treatment is resorted to, the material should be soaked for a day or more in the clearing reagent, and examined in the same medium on the slide.

The presence of occasional traces of a foreign substance, when viewed under the microscope, is hardly sufficient to condemn the sample as adulterated. Unless they are evident by being persistently apparent in a large number of fields, such traces are apt to be accidental.

Composition of Miscellaneous Spice Adulterants.— The chemical analyses of various spice adulterants commonly met with are given on page 315.

CLOVES.

Nature and Composition.—Cloves are the dried, undeveloped flowers of the clove tree (*Caryophyllus aromaticus* or *Eugenia caryophyllata*), which belongs to the myrtle family (*Myrtaceæ*). The tree is an evergreen, from twenty to forty feet in height, cultivated extensively in Brazil, Cey-

	Moisture.	Ash.			Ether Extract.		Alcohol Extract.
		Total.	Soluble in Water.	Insoluble in HCl.	Volatile.	Non-volatile.	
English-walnut shells *	7.69	1.40	0.77	0.00	0.12	0.55	1.84
Brazil-nut shells *	9.08	1.59	1.06	0.17	0.07	0.57	1.01
Almond shells *	7.80	2.86	2.39	0.05	0.16	0.64	5.16
Cocoanut shells *	7.36	0.54	0.50	0.00	0.00	0.25	1.12
Date stones *	8.24	1.24	0.76	0.04	0.36	8.38	16.72
Spruce sawdust *	8.77	0.23	0.16	0.00	0.07	0.77	1.50
Oak sawdust *	5.73	1.22	0.32	0.02	0.07	0.84	6.25
Linseed meal *	8.71	5.72	1.74	0.55	0.04	6.58	9.46
Cocoa shells *	10.44	8.40	4.66	0.83	1.00	2.99	4.77
Red sandalwood *	4.42	0.70	0.28	0.07	1.21	11.47	19.37
Ground olive stones †	9.50	0.88	0.24	0.44	0.06	0.24	

	Reducing Matters by Acid Conversion.	Starch by Diastase Method.	Crude Fiber.	Nitrogen × 6.25.	Oxygen Absorbed by Aqueous Extract.	Quercitannic Acid Equivalent.	Total Nitrogen.
English-walnut shells *	19.30	1.01	56.58	1.69	0.53	2.08	0.27
Brazil-nut shells *	12.96	0.73	50.98	4.19	0.33	1.30	0.67
Almond shells *	22.72	0.84	49.89	1.75	0.40	1.56	0.28
Cocoanut shells *	20.88	0.73	56.19	1.13	0.47	1.82	0.18
Date stones *	20.88	2.19	5.72	5.31	0.61	2.34	0.85
Spruce sawdust *	15.48	1.13	64.03	0.56	0.30	1.17	0.09
Oak sawdust *	17.10	1.68	47.79	1.63	3.13	12.22	0.26
Linseed meal *	21.15	14.06	8.30	31.81	1.00	3.90	5.09
Cocoa shells *	8.68	3.15	14.12	16.19	1.26	4.94	2.59
Red sandalwood *	6.79	1.12	52.30	3.06	0.59	2.29	0.49
Ground olive stones †	.....	1.73	57.46	1.06	.....	.....	0.17

75 per cent of the oil, and a sesquiterpene known as caryophyllene. There are also in cloves a notable amount of fixed oil and resin, and also a peculiar form of tannin.

Very few complete analyses of cloves are on record. Richardson ‡ seems to have been the earliest worker in the field to give anything at all satisfactory in the way of a number of determinations of value.

The following are maximum and minimum figures from the tabulated results of Richardson's analyses:

* Winton, Ogden, and Mitchell, Conn. Exp. Sta. An. Rep., 1898, p. 210.

† Doolittle, Mich. Dairy and Food Dept. Bul. 94, 1903, p. 12.

‡ U. S. Dept. of Agric., Div. of Chem., Bul. 13.

	Water.	Ash.	Volatile Oil.	Fixed Oil and Resin.	Crude Fiber.	Albuminoids.	Nitrogen.	Oxygen Equivalent.	As Quercitannic Acid.
Whole cloves (7 samples):									
Maximum	10.67	13.05	18.89	10.24	9.75	7.	1.12	5.43	22.13
Minimum	2.90	5.50	10.23	7.12	6.18	4.73	.76	3.00	11.70
Stems (1 sample)	10.18	6.96	4.40	4.03	13.58	5.78	.92	5.96	23.24
Ground cloves (9 samples):									
Maximum	9.58	10.73	13.93	7.44	13.80	6.48	1.04	6.20	24.18
Minimum	5.93	5.79	3.94	4.02	9.38	4.20	.70	2.89	11.28

McGill * gives tables of analyses of pure and adulterated samples of cloves. Analyses of upwards of twenty samples of genuine cloves, both whole and ground, from these tables show the following maximum and minimum figures:

	Maximum.	Minimum.
Moisture	11.80	5.05
Volatile oil	19.63	9.24
Total volatile matter	30.68	16.25
Fixed oil	10.23	0.94
Total extraction	31.40	22.23
Ash	7.00	5.03

McGill also made analyses of whole cloves of several varieties, the following table being a summary of his results:

	No. of Analyses.	Moisture.	Total Volatile Matter.	Volatile Oil.	Total Extractive Matter.	Fixed Oil.
Penang cloves: Maximum	8	7.4	24.3	17.2	28.2	12.0

	Maximum.	Minimum.
Ash, total	7.92	5.99
Ether extract, volatile	18.25	11.03
" " non-volatile	7.19	4.87

Winton, Ogden, and Mitchell * give more complete analyses of eight samples of whole cloves of known purity, and two samples of clove stems as follows:

	Moisture.	Ash.			Ether Extract.		Alcohol Extract.
		Total.	Soluble in Water.	Insoluble in HCl.	Volatile.	Non-volatile.	
Maximum	8.26	6.22	3.75	0.13	20.53	6.67	15.58
Minimum	7.03	5.28	3.25	0.00	17.82	6.24	13.99
Mean	7.81	5.92	3.58	0.06	19.18	6.40	14.87
Clove stems, mean	8.74	7.99	4.26	0.60	5.00	3.83	6.79

	Reducing Matters by Acid Conversion, as Starch.	Starch by Diastase Method.	Crude Fiber.	Nitrogen, ×6.25.	Oxygen Absorbed by Aqueous Extract.	Quercitannic Acid.	Total Nitrogen.
Maximum	9.63	3.15	9.02	7.06	2.63	20.54	1.13
Minimum	8.19	2.08	7.06	5.88	2.08	16.25	0.94
Mean	8.99	2.74	8.10	6.18	2.33	18.19	0.99
Clove-stems, mean	14.13	2.17	18.71	5.88	2.40	18.79	0.94

The Tannin Equivalent in Cloves.—The amount of tannin in cloves was shown by Ellis to be so constant as to be of valuable assistance as a guide to their purity. The actual determination of tannin is, however, a long and difficult proceeding, and Richardson † has pointed out that it is not necessary, but that simply using the first part of the Lowenthal tannin process, and noting the "oxygen absorbed" as expressed by the oxidizing power of permanganate of potash on the material after extraction with ether, is quite as useful as determining the tannin, and is in effect proportional to the tannin present. The result is sometimes expressed as in Richardson's figures above, as the *oxygen equivalent*, or as *quercitannic acid*.

Determination of Tannin Equivalent.‡—*Reagents: Indigo Solution.*—Six grams of the indigo salt § are dissolved in 500 cc. of water by heat-

* Conn. Exp. Sta. Rep., 1898, pp. 206, 207.

† U. S. Dept. of Agric., Div. of Chem., Bul. 13, p. 167.

‡ Winton, U. S. Dept. of Agric., Bur. of Chem., Bul. 65, p. 60.

§ The quality of the indigo used is of great importance since with inferior brands it is

ing. After cooling, 50 cc. of concentrated sulphuric acid are added, the solution made up to a liter and filtered.

Standard Permanganate Solution.—Dissolve 1.333 grams of pure potassium permanganate in a liter of water. This should be standardized by titrating against 10 cc. of tenth-normal oxalic acid (6.3 grams pure crystallized oxalic acid in 1,000 cc.), diluted to 500 cc. with water, heated to 60° C., and mixed with 20 cc. of dilute sulphuric acid (1:3 by volume). The permanganate solution is added slowly, stirring constantly, till a pink color appears.

Two grams of the material are extracted for twenty hours with pure anhydrous ether. The residue is boiled for two hours with 300 cc. of water, cooled, made up to 500 cc., and filtered.

Twenty-five cc. of the filtrate are pipetted into a 1200-cc. flask, 750 cc. of distilled water are added and 20 cc. of indigo solution.

The standard permanganate solution is then run in from a burette a drop at a time with constant shaking, until a bright golden yellow color appears, which indicates the end-point. Note the number of cubic centimeters required, represented by (*a*).

In a similar manner determine the number of cubic centimeters of standard permanganate solution consumed by 20 cc. of the indigo solution alone, represented by (*b*), and subtract this from (*a*).

The oxygen equivalent, or, as it is sometimes called, the "oxygen absorbed," is calculated from the equivalent in tenth-normal oxalic acid of the number of cubic centimeters of standard permanganate represented by *a*—*b*. 10 cc. of tenth-normal oxalic acid are equivalent to 0.008 gram of oxygen absorbed, or 0.0623 gram of quercitannic acid.

Microscopical Examination of Cloves.—Unless the finely powdered, water-mounted sample is well rubbed out under the cover-glass, many



surrounded by irregularly shaped cells; (3) represents the epidermis of the petals, with crystals of calcium oxalate; a cross-section of the epidermis of the calyx is shown at (4); (5) shows the parenchyma, with calcium oxalate crystals and with one of the slender spiral ducts; (6) and (7) represent in cross-section and longitudinal section respectively the parenchyma of the middle layers of the calyx, one of the rounded, triangular pollen grains being shown at (12).

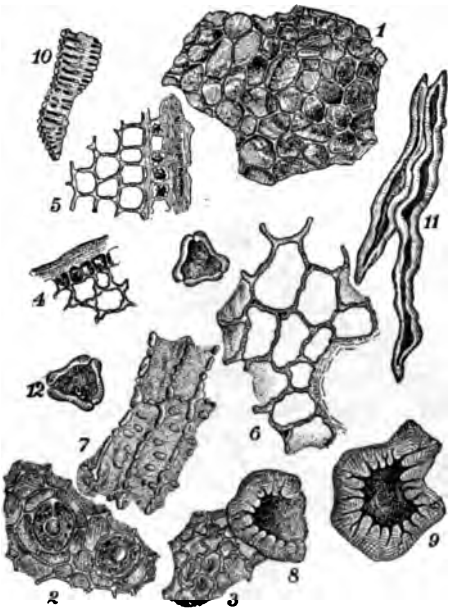


FIG. 74.—Powdered Cloves under the Microscope. ×125. (After Moeller.)

Characteristics of clove stems, which are frequently used as adulterants of cloves, are found in (8), (9), (10), and (11). Stone cells of the outer skin and the inner portion of the clove stem are shown at (8) and (9) respectively; (10) shows one of the vascular ducts, and (11) two of the bast fibers. Both the vascular ducts and the bast fibers are very characteristic of clove stems. Pure cloves have no stone cells and comparatively few bast fibers. Stems under the microscope show a large number of bast fibers and frequent stone cells.

A plain water-mounted slide rarely shows all the structural details depicted in Fig. 74, but is nearly always sufficiently characteristic to

prove the purity of the sample. Fig. 220, Pl. XXV, shows the actual appearance of powdered cloves, mounted in water and examined under a magnification of 130. The general appearance of the cellular tissue is that of a loose, spongy mass filled with brown, granular material. Throughout the masses of tissue are to be seen small oil globules.

Cloves have no starch whatever. Aside from the stems, cloves are sometimes adulterated with clove fruit or "mother cloves," which have a small amount of a sago-like starch, and also contain some stone cells.

Adulteration of Cloves.—The U. S. standard for pure cloves is as follows: Volatile ether extract not less than 10%; quercitannic acid, calculated from the total oxygen absorbed by the aqueous extract, should not be less than 12%; total ash should not exceed 8%; ash insoluble in hydrochloric acid should not exceed 0.5%, and crude fiber should not be more than 10%.

Clove Stems are very frequent adulterants of cloves and possess some slight pungency. They are commonly identified under the microscope by the large number of bast fibers and stone cells, and should not be found in pure cloves in excess of 5%.

Allspice, being considerably cheaper than cloves, is sometimes used as an adulterant. It is readily recognized by the characteristics described on page 324.

Other Adulterants commonly found are cereal starches (especially corn and wheat) and ginger (for the most part "exhausted"). Besides the above, pea starch, rice, turmeric, charcoal, sand, pepper, ground fruit stones, and sawdust have been found in samples of cloves examined in Massachusetts.

Exhausted Cloves, both whole and in powdered form, are not infre-



With the exception of exhausted cloves, the presence of nearly every foreign ingredient is best and most quickly shown by the use of the microscope, though much information as to the purity of the sample can be gained by the ether extract, the percentage of ash, and of crude fiber.*

Cocoanut Shells.—Figs. 226 and 227, Pl. XXVII, show samples of cloves adulterated with ground cocoanut shells. The long, spindle-shaped, yellow-brown and deeply furrowed stone cells of the adulterant with their thick walls and central branching pores are unmistakable. The dark-brown contents of the cells turn reddish brown when treated with potassium hydroxide. The anatomy of the cocoanut, including the shell, has been carefully studied by Winton.†

Fig. 75, after Winton, shows elements of powdered cocoanut shell under the microscope. *st* are the dark, elongated, yellow, porous stone

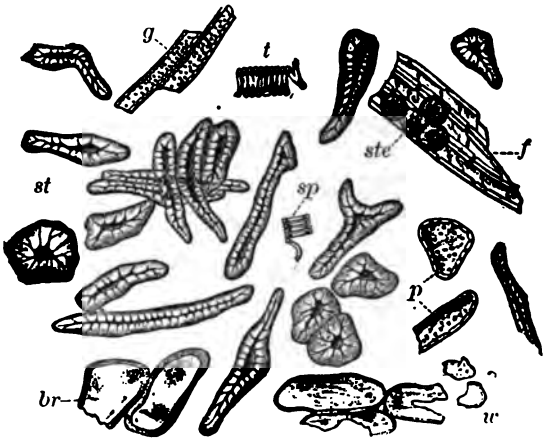


FIG. 75.—Cocoanut-shell Powder. *st*, dark-yellow stone cells with brown contents; *t*, reticulated trachea; *sp*, spiral trachea; *g*, pitted trachea; *w*, colorless, and *br*, brown, parenchyma of mesocarp; *f*, bast fibres, with stigmata (*ste*). ×160. (After Winton.)

cells with their brown contents, these stone cells being the most distinctive characteristic of the ground shells. *t*, *sp*, and *g* are the various forms of trachea; *w* and *br* are respectively colorless and brown parenchyma of the mesocarp or outer coat, portions of which always adhere to the nutshell and are ground with it.

* Note especially the sharp distinction between these values in the case of pure cloves and of clove stems in Richardson's table.

† The Anatomy of the Fruit of the Cocoanut. Conn. Exp. Sta. Rep., 1901, p. 208.

Fig. 264, Pl. XXXVI, shows a photomicrograph of powdered cocoanut shells mounted in gelatin. The long, spindle-shaped stone cells are especially apparent.

Cocoanut shells are very common adulterants of various spices besides cloves, especially of allspice and pepper. In the following tabulated results of analyses by Winton, Ogden, and Mitchell * are shown the wide deviation between the chemical constants of cocoanut shells and several of the spices in which they appear as adulterants.

	Black Pepper.	Cloves.	Allspice.	Nutmeg.	Cocoanut Shells.
Water	11.96	7.81	9.78	3.63	7.36
Total ash	4.76	5.92	4.47	2.28	0.54
Ash soluble in water	2.54	3.58	2.47	0.86	0.50
Ash insoluble in hydrochloric acid	0.47	0.06	0.03	0.00	0.00
Volatile ether extract	1.14	19.18	4.05	3.02	0.00
Non-volatile ether extract	8.42	6.49	5.84	36.70	0.25
Alcohol extract	9.62	14.87	11.79	10.77	1.12
Reducing matters, as starch, acid conversion	38.63	8.99	18.03	25.56	20.88
Starch by diastase method	34.15	2.74	3.04	23.72	0.73
Crude fiber	13.06	8.10	22.39	2.51	56.19
Total nitrogen	2.26	0.99	0.92	1.08	0.18
Oxygen absorbed by aqueous extract	.....	2.33	1.24	.....	0.23
Quercitannic acid equivalent	.....	18.19	9.71	.....	1.83

ALLSPICE, OR PIMENTO.

Nature and Composition.—Allspice is the dried fruit of the *Eugenia pimenta*, an evergreen tree belonging to the same family (*Myrtaceæ*) as the clove. It is indigenous to the West Indies, and is especially cultivated in Jamaica.

The allspice berry is grayish or reddish brown in color, and is hard and globular, measuring from 4 to 6 mm. in diameter, being surmounted

Though considerably less pungent than other spices, allspice possesses an aroma not unlike cloves and cassia. In chemical composition it most resembles cloves, containing both volatile oil and tannin; but, unlike cloves, it contains much starch, the starch being contained in the seeds. The volatile oil of allspice is very similar to clove oil. It is slightly lævorotary, and is composed of eugenol and a sesquiterpene not determined. It is present in allspice to the extent of 3 to 4.5 per cent. The boiling-point of the oil is 255° C.

Authoritative full analyses of allspice are even more meager than of cloves. Analyses of one sample of whole allspice and five samples of the ground spice, made by Richardson,* are thus summarized:

	Water.	Ash.	Volatile Oil.	Fixed Oil.	Undetermined.	Crude Fiber.	Albuminoids.	Nitrogen.	Tannin Equivalent.	Oxygen. Required.
Whole	6.19	4.01	5.15	6.15	59.28	14.83	4.38	.70	10.97	2.81
Ground:										
Maximum	8.82	5.53	3.32	6.92	58.24	23.60	5.42	.87	12.74	3.36
Minimum	5.51	3.45	1.29	1.60	55.90	13.45	4.03	.67	4.32	1.11

Seventeen samples of unadulterated allspice, as sold on the Connecticut market, were analyzed by Winton and Mitchell,† with maximum and minimum results as follows:

Ash.	Maximum.	Minimum.
Total	7.51	4.34
Insoluble in hydrochloric acid (sand)	.95	.40
Ether extract, volatile	3.50	1.34
Ether extract, non-volatile	6.22	3.78

Three samples of pure whole allspice were more fully analyzed by Winton, Mitchell, and Ogden with the results given on page 324.‡

The Tannin Equivalent in Allspice.—Tannin is present in allspice, though to a less extent than in cloves. The exact amount present is rarely determined, but rather the "oxygen equivalent," or quercitannic acid, as explained on page 317, the determination being carried out as there detailed.

* U. S. Dept. of Agric., Div. of Chem., Bul. 13, p. 229.

† An. Rep. Conn. Exp. Sta., 1898, pp. 178, 179.

‡ *Ibid.*, pp. 208, 209.

	Moisture.	Ash.			Ether Extract.		Alcohol Extract.
		Total.	Soluble in Water.	Insoluble in HCl.	Volatile.	Non-volatile.	
Maximum	10.14	4.76	2.69	0.06	5.21	7.72	14.27
Minimum	9.45	4.15	2.29	0.00	3.38	4.35	7.39
Average	9.78	4.47	2.47	0.03	4.05	5.84	11.79

	Reducing Matters by Acid Conversion, as Starch.	Starch by Diastase.	Crude Fiber.	Nitrogen, ×6.25.	Oxygen Absorbed by Aqueous Extract.	Quercitannic Acid.	Total Nitrogen.
Maximum	20.65	3.76	23.98	6.37	1.59	12.48	1.02
Minimum	16.56	1.82	20.46	5.19	1.03	8.06	0.83
Average	18.03	3.04	22.39	5.75	1.24	9.71	0.92

Microscopical Examination of Powdered Allspice.—By soaking the powder twenty-four hours or more in chloral hydrate, many of the harder portions are rendered much more transparent than would otherwise be possible. Fig. 76, after Moeller, shows the microscopical structure of various elements that go to make up allspice powder.

The epidermis, or outer layer of the berry, is shown at (1) in cross-section, and in plan view at (2) with its small cells. Just beneath the outer coat are the large oil spaces (1*b*) and still further below the stone-cells (1*c*). The fruit parenchyma (3) has vascular tissues running through it. (4) and (5) are the inner epidermis and stone cells of the dividing partitions between the seeds. Small hairs connected with the outer epidermis are shown at (6). (7) and (8) show in cross-section a portion of the seed-shell and inclosed seed or embryo, with the starch (8*a*) and the colored lumps of gum or resin (8*b*) of a port-wine color. These colored



Three distinctive features are especially typical, viz.: First, the starch grains, which are very uniform in size, measuring about 0.008 mm. in diameter, being nearly circular as a rule, and often arranged in groups not unlike masses of buckwheat starch. Ordinarily these masses contain fewer granules than do those of buckwheat. The granules are

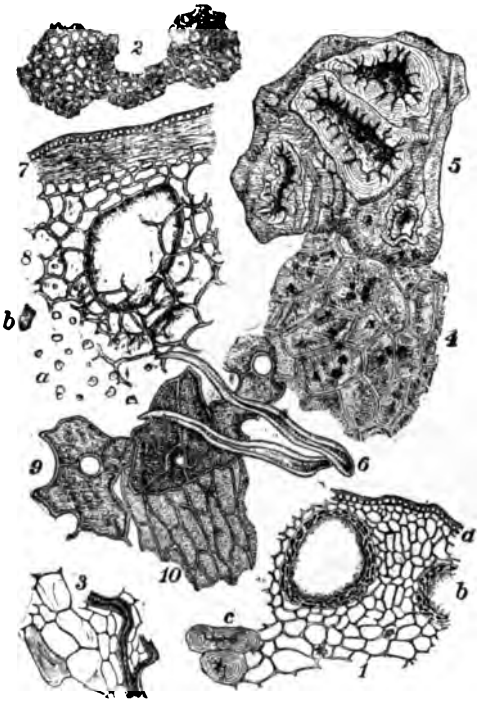


FIG. 76.—Powdered Allspice under the Microscope. ×125. (After Moeller.)

smaller and more inclined to the circular than to the polygonal form, while in many cases they have distinct central hila. The starch grains are very numerous and are found in nearly every field. See Fig. 195, Pl. XIX.

A second distinctive feature of allspice is the stone cells, of which there are many. These are more often colorless, and in most cases very large and plainly marked. They are sometimes seen singly and at other times grouped together. Frequently they are attached to pieces of brown parenchyma.

The third and most characteristic feature of allspice powder under the microscope is the striking appearance of the lumps of gum or resin, which are cells of a more or less deep port-wine or amber color. These are contained in the delicate epidermis of the seed coat, and are very striking, occurring sometimes in isolated bits, and in other cases in aggregations of from 2 to 4 or even 6 to 8 cells. These resinous lumps appear plainly in Fig. 194, Pl. XIX. Droplets of oil are occasionally seen, but not in profusion. As a rule the oil is forced out of its large containing cells and into the surrounding tissue by the process of drying.

Adulteration of Allspice.—According to the U. S. standard for allspice, quercitannic acid should not be less than 8%, total ash not more than 6%, ash insoluble in hydrochloric acid not more than 0.5%, crude fiber not more than 25%. The most common adulterants found in powdered allspice are cocoanut shells and the cereal starches. Besides these the writer has found in Massachusetts, peas, pea hulls, exhausted ginger, cayenne, olive stones, pepper, and turmeric. To this list may be added clove stems, which are on record as a not uncommon adulterant in some localities. All of these are to be readily recognized by a careful microscopical examination.

CASSIA AND CINNAMON.

Nature and Composition. — The terms cassia and cinnamon are interchangeable in commerce, though, strictly speaking, they represent two separate and distinct species of the genus *Cinnamomum*, belonging to the laurel family (*Lauraceæ*). True cinnamon is the bark of *Cinnamomum zeylanicum*, a tree from 20 to 30 feet high, having horizontal



that it is of a much cleaner and more even texture than the cassia bark, which is thicker and heavier by reason of the outer cork layer usually left on it.

The cheaper and more common cassia is the bark of the *Cinnamomum cassia*, which comes from China, Indo-China, and India. It is of a darker color than that of cinnamon, of coarser texture, and as a rule about four times as thick. Most varieties of cassia bark are less tightly rolled than cinnamon, and are not arranged one within the other in layers. The outer surface is marked by elliptical spots left by the leaves, and by small, dark-brown, wart-like protuberances. Cassia does not have the wavy, light-colored lines found in the cinnamon. Both cinnamon and cassia barks are very aromatic in taste, somewhat astringent, and slightly sweet.

Cassia buds are the dry flower buds of China cassia, and are found in the market both in whole and in powdered form. Powdered cassia often consists of a mixture of several varieties of bark, while the cheaper grades sometimes contain an admixture of the ground buds.

The best grade of cassia is that from Saigon, while the cheapest is the Batavia cassia.

The odor of cassia and cinnamon bark is due to the volatile oil, of which from 1 to 2 per cent is usually found. Cassia and cinnamon oil greatly resemble each other, the principal constituent in either case being cinnamic aldehyde, $C_6H_5CH:CH.CHO$. Besides this, one or more esters of acetic acid are present. Both oils are very pungent and intensely sweet.

Starch is present in cassia to the extent of from 16 to 30 per cent. A very small amount of tannin is found, as well as cinnamic acid and mucilaginous matters. Cassia buds are somewhat similar in composition to the bark. They have, however, less starch and crude fiber, and higher contents of volatile oil and nitrogen than the bark.

Richardson * has made analyses of a few samples of pure whole cinnamon and cassia, from which the following are taken:

	Water.	Ash.	Volatile Oil.	Fixed Oil, etc.	Crude Fiber.	Albuminoids.	Undetermined.	Nitrogen.
Ceylon cinnamon 1.....	5.40	4.55	1.05	1.66	33.08	2.98	51.28	.48
" " 2.....	7.43	3.40	.82	1.58	25.63	3.80	56.84	.62
Cassia buds.....	4.79	5.58	3.59	5.21	8.60	7.00	65.23	1.12
Cassia bark (4 samples):								
Maximum.....	17.45	8.23	3.51	2.13	26.29	4.55	65.33	.73
Minimum.....	9.32	2.48	.55	.74	14.33	2.63	48.65	.42

* U. S. Dept. of Agric., Div. of Chem., Bul. 13, p. 221.

Winton, Ogden, and Mitchell's* results of analyses of whole samples of cinnamon, cassia, and cassia buds are thus summarized:

	Moisture.	Ash.			Ether Extract.	
		Total.	Soluble in Water.	Insoluble in HCl.	Volatile.	Non-volatile.
Ceylon cinnamon (6 samples):						
Maximum	10.48	5.99	2.71	0.58	1.62	1.68
Minimum	7.79	4.16	1.40	0.02	0.72	1.35
Average	8.63	4.82	1.87	0.13	1.39	1.44
Cassia bark (20 samples):						
Maximum	11.91	6.20	2.52	2.42	5.15	4.13
Minimum	6.53	3.01	0.71	0.02	0.93	1.32
Average	9.24	4.73	1.68	0.56	2.61	2.12
Cassia buds (2 samples):						
Average	7.93	4.64	2.88	0.27	3.88	5.96

	Alcohol Extract.	Reducing Matters by Acid Conversion, as Starch.	*Crude Fiber.	Nitrogen, ×6.25.	Total Nitrogen.
Ceylon cinnamon (6 samples):					
Maximum	13.60	22.00	38.48	4.06	0.65
Minimum	9.97	16.65	34.38	3.25	0.52
Average	12.21	19.30	36.20	3.70	0.59
Cassia bark (20 samples):					
Maximum	16.74	32.04	28.80	5.44	0.87
Minimum	4.57	16.65	17.03	3.31	0.53
Average	8.29	23.32	22.96	4.34	0.69
Cassia buds (2 samples):					
Average	10.88	10.71	13.35	7.53	1.20

Structure of Powdered Cassia under the Microscope. — Fig. 77, from Moeller, shows various elements of cassia bark as veiwed microscopically. (1) shows in cross-section a portion of the cork and outer layer



and inner bark layers. (6) represents a fragment of a bast fiber, which is often shown in cassia powder with connecting parenchyma. The stone-cells of the cork are shown in plan view at (7). Very small, needle-like crystals of oxalate of calcium are occasionally to be seen if looked for carefully. They occur in the parenchyma cells of the inner and intermediate layers of the bark.

The microscopical structure of Ceylon cinnamon much resembles that of cassia. Cassia starch grains measure from 0.0132 to 0.0222 mm.,

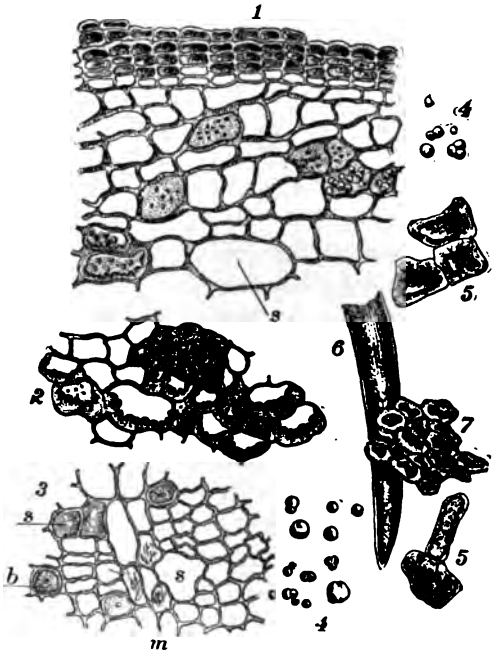


FIG. 77.—Powdered Cassia under the Microscope. ×125. (After Moeller.)

being considerably larger and more abundant that those of true cinnamon. As a rule the bast fibers of cassia are larger, but shorter, than those of cinnamon, and provided with thicker walls.

Figs. 203 and 204, Pl. XXI, show various phases of pure cassia bark as photographed from water-mounted specimens of the powder. Cassia starch somewhat resembles that of allspice, but it is not as a rule found in masses containing as many granules as does the allspice starch. Very commonly two or three of the starch granules are arranged together in

such a manner that at first sight they appear to form a single large granule, but on more careful examination are seen to be two- and three-lobed, consisting of several smaller grains. Stone cells, which are very abundant in the powdered cassia, do not happen to be included to any extent in the photographed fields. Cassia stone cells are generally more oblong than those of allspice, and are more often brown in color, while the allspice stone cells are generally colorless.

A distinctive feature of powdered cassia consists in the long, amber-colored wood fibers, some distributed in bundles, and others arranged singly. These are very clearly shown in Figs. 204 and 205.

Yellow patches of cellular tissue with starch grains interspersed among them are very abundant in the powder.

Adulteration of Cinnamon and Cassia.—The U. S. standards for cassia and cinnamon are as follows: Total ash not to exceed 8%; sand not to exceed 2%.

The adulterants most commonly found in these products are the cereal starches and ground foreign bark. Besides these, the writer has found, in the natural course of inspection in Massachusetts, leguminous starches, pea hulls, nutshells, turmeric, pepper, olive stones, ginger, mustard, and sawdust.

Ground Bark of the Common Trees, especially that of the elm, resembles in physical appearance ground cassia, and is to be looked for as an adulterant. Fig. 265, Pl. XXXVII, shows the appearance of ground elm bark. The fibers of cassia bark have starch granules as a rule interposed among them, while the foreign bark, usually of a much coarser texture, shows no starch connected with its structure.

Fig. 206, Pl. XXII, shows a water-mounted specimen of adulterated [redacted]

eter, covered with a brownish-gray epicarp, and having on the under side the remains of a short stem. At the top of the berry is an indistinct trace of a style, and of a lobed stigma.

Varieties of black pepper are named from the localities in which they grow, as Singapore, Lampong, Sumatra, Tellichery, Malabar, Acheen, Penang, Allepey, Trang, etc.

White pepper is obtained by decorticating the fully ripened black peppercorns, or removing the dark skin. This is accomplished by macerating them in water to loosen the skin, which is then removed readily by drying and rubbing between the hands. White whole pepper grains are grayish white, and a trifle larger than the black pepper berries. They are nearly spherical in shape, and have a number of light-colored lines that, like meridians, run from top to bottom.

Varieties of whole white natural pepper are obtained from Siam and Singapore, but are not frequently met with on the market.

The pungent taste of pepper is due in great part to its essential oil, a hydrocarbon of the formula $C_{10}H_{16}$, present in amounts varying from 0.5 to 1.7 per cent. Pepper oil contains phellandrene and a terpene.

Another important constituent of pepper, contributing to its pungency, is the crystalline base *piperin*, $C_{17}H_{19}NO_3$, insoluble in water, but soluble in ether, and in alcohol. Starch is present in pepper to a large extent.

Burcker gives the following average percentage composition of black and white pepper:

	Ash.	Cellulose.	Water.	Nitrogenous Matter.	Volatile Oil.	Fat, Piperin, and Resin.	Starch and Dextrin.	Other Non-nitrogenous Matters.
Black pepper.....	4.57	12.45	12.50	11.98	1.36	6.85	42.90	7.39
White pepper.....	1.80	6.08	13.56	11.12	0.94	7.11	56.04	3.35

Richardson's * analyses of three samples of whole black and two samples of whole white pepper, all pure, are as follows:

		Water.	Ash.	Volatile Oil.	Piperin and Resin.	Alcohol Extract.	Starch.
Black pepper:	West coast.....	8.91	4.04	.70	7.29	....	36.52
	Acheen.....	8.29	4.70	1.69	7.72	6.06	37.50
	Singapore.....	9.83	3.70	1.60	7.15	5.74	37.30
White pepper:	West coast.....	9.85	1.41	.57	7.24	....	40.61
	Singapore.....	10.60	1.34	1.26	7.76	2.57	43.10

* U. S. Dept. of Agric., Bur. of Chem., Bul. 13, part 2, p. 206.

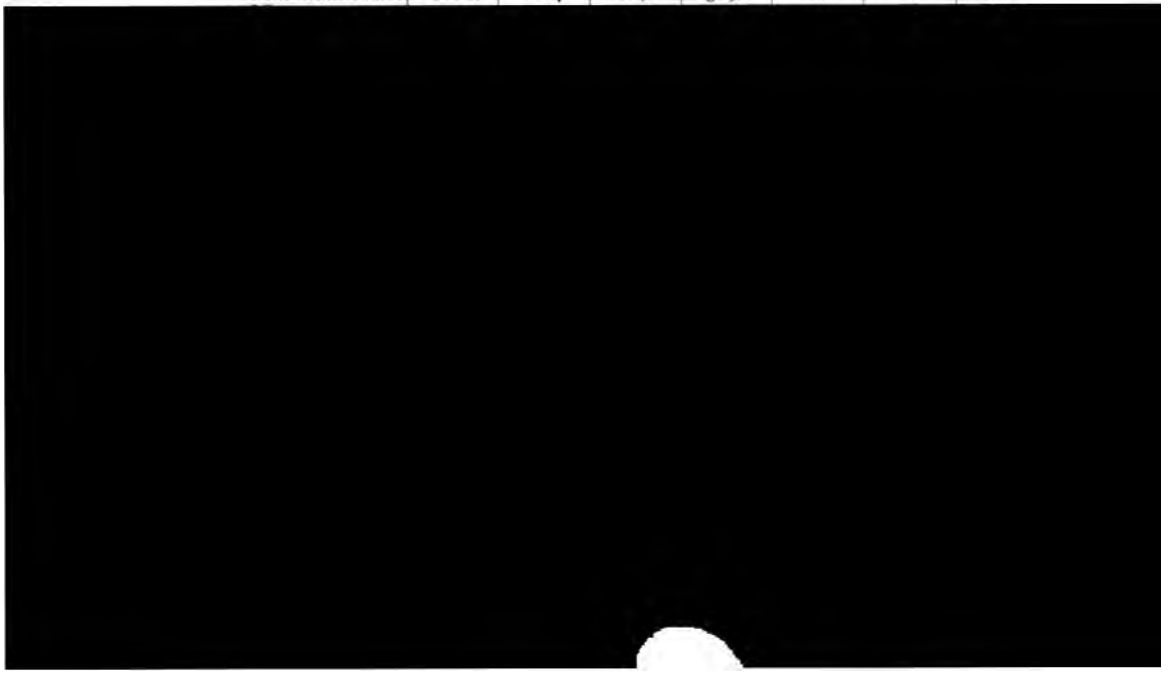
		Undetermined.	Crude Fiber.	Albuminoids.	Total N×6.25.	Total N.
Black pepper:	West coast.	24.62	10.23	7.69	9.81	1.57
	Acheen.	13.64	10.02	10.38	12.60	2.02
	Singapore	17.66	10.02	10.00	12.08	1.93
White pepper:	West coast.	23.28	7.73	9.31	11.48	1.83
	Singapore	19.55	4.20	9.62	11.90	1.90

Richardson gives the following variations in the constituents of pure pepper:

	Black.	White.
Water	8.0 to 11.0	8.0 to 11.0
Ash	2.75 to 5.0	1.0 to 2.0
Volatile oil	.50 to 1.75	.50 to 1.75
Piperin and resin	7.0 to 8.0	7.0 to 8.0
Starch	32.0 to 38.0	40.0 to 44.0
Crude fiber	8.0 to 11.0	4.11 to 8.0
Albuminoids	7.0 to 12.0	8.0 to 10.0

McGill's * analyses of six samples of whole black, and five samples of whole white pepper, all genuine, are thus summarized:

	Moisture, etc., Lost at 100° C.	Ash.					Alcohol Extract.
		Soluble in Hot Water.	Insoluble in Water.	Total.	Insoluble in Hydrochloric Acid.	Sand Expressed as Per Cent of Total Ash.	
Black: Maximum	14.10	2.64	3.06	5.16	1.08	21	9.06
Minimum	10.62	2.07	1.46	3.98	.06	2	8.28



	Moisture.	Ash.			Ether Extract.		Alcohol Extract.
		Total.	Soluble in Water.	Insoluble in HCl.	Volatile.	Non-volatile.	
Black pepper: Maximum	12.95	6.52	3.20	1.19	1.60	10.37	11.86
Minimum.	10.63	3.09	1.75	0.00	0.65	6.86	8.47
Average	11.96	4.76	2.54	0.47	1.14	8.42	9.62
White pepper: Maximum	14.47	2.96	0.80	0.20	0.95	7.94	8.55
Minimum.	12.72	1.03	0.28	0.00	0.49	6.26	7.19
Average	13.47	1.77	0.47	0.10	0.73	6.91	7.66
Pepper shells: Maximum	10.66	11.91	3.20	4.70	1.06	4.97	6.30
Minimum.	10.52	10.25	2.28	2.63	0.68	3.04	4.00
Long pepper	9.47	5.93	4.20	0.22	1.55	6.61	8.67
Buckwheat hulls	7.63	1.84	1.24	0.00	0.07	0.38	2.17

	Reducing Matters by Acid Conversion, as Starch.	Starch by Diastase.	Crude Fiber.	Total Nitrogen less N in Ether Extract×6.25.	Nitrogen.	
					Total.	In non-volatile Ether Extract.
Black pepper: Maximum	43.47	39.66	18.25	13.81	2.53	0.40
Minimum.	28.15	22.05	10.75	10.50	2.03	0.27
Average	38.63	34.15	13.06	13.06	2.26	0.33
White pepper: Maximum	64.92	63.60	4.25	11.19	2.13	0.34
Minimum.	56.43	53.11	0.54	10.44	1.95	0.26
Average	59.17	56.47	3.14	10.89	2.04	0.30
Pepper shells: Maximum	21.69	15.30	32.15	14.19	2.36	0.15
Minimum.	11.43	2.30	23.27	12.31	2.12	0.09
Long pepper	42.88	39.55	5.76	12.25	2.18	0.22
Buckwheat hulls	20.51	1.46	43.76	3.06	0.49	

Winton and Mitchell's analyses of ground pepper, sold in labeled packages in Connecticut and pronounced unadulterated, are as follows:

	Ash.		Non-volatile Ether Extract.	Crude Fiber.
	Total.	Insoluble Hydrochloric Acid.		
Black, 36 samples: Maximum	7.61	5.21	9.37	17.09
Minimum.	3.90	....	6.54	7.64
White, 12 samples: Maximum	4.01	....	8.27	6.49
Minimum.	1.09	....	4.07	0.43

The following table summarizes the results of full analyses of pepper and pepper shells recently made by Doolittle: *

* Mich. Dairy and Food Comm Bul. 94.

	No. of Samples.	No. of Varieties.	Moisture.	Ash. Total.	Ash. Insoluble in HCl.	Ash. Soluble in Water.	Starch by Diastase Method.
Black pepper:	45	12					
Maximum	....	....	11.96	8.04*	1.52*	3.32	42.75
Minimum	....	....	8.20	3.43	0.05	1.05	25.00
Average	....	....	9.54	4.90	0.58	2.46	36.00
White pepper:	25	9					
Maximum	....	....	13.34	4.26	0.86	1.16	63.55
Minimum	....	....	8.24	0.86	0.05	0.12	48.88
Average	....	....	9.87	1.96	0.13	0.54	54.37
Long pepper:	3						
Maximum	....	....	10.13	14.30	5.32	4.30	45.87
Minimum	....	....	8.43	0.12	0.45	1.72	28.43
Pepper shells:	4						
Maximum	....	....	11.01	28.82	22.30	4.56	11.70
Minimum	....	....	7.00	7.82	0.79	1.53	1.28

	Ether Extract. Volatile.	Ether Extract. Non-volatile.	Crude Fiber.	Nitrogen. Total.	Nitrogen. In Non-volatile Ether Extract.	Total N less N in Non-volatile Ether Extract (Protein)
Black pepper:						
Maximum	2.12	10.44	18.80	2.58	0.45	13.11
Minimum	0.85	0.80	10.05	1.80	0.25	7.25
Average	1.30	7.07	11.12	2.11	0.32	11.20
White pepper:						
Maximum	1.66	7.26	7.05	2.14	0.34	11.52
Minimum	0.78	5.05	0.12	1.85	0.14	1.06
Average	1.17	6.42	4.17	1.97	0.30	10.44
Long pepper:						
Maximum	2.22	7.33	12.12	2.24	0.32	12.06
Minimum	0.75	5.72	7.12	2.13	0.28	11.37
Pepper shells:						
Maximum	2.12	4.17	28.22	1.82	0.12	11.25
Minimum	0.81	1.52	11.20	1.72	0.12	10.20

*The specimen of Ashanti Cayenne had a total ash of 8.04% and 6.08% acid-insoluble ash.

sulphate, and 25 cc. of sulphuric acid in a Kjeldahl flask, for both digestion and distillation, of about 600-cc. capacity. The heating is conducted in the usual manner, beginning with a gentle heat till the frothing ceases, and gradually increasing the temperature till the mixture boils. The boiling is continued for three or four hours, after which the flask is cooled, and to it are added 300 cc. of water, 50 cc. of potassium sulphide solution,* and enough of a saturated solution of sodium hydroxide to render the reaction alkaline.

The flask is then connected to the condenser, and the distillation conducted as in the Gunning method (p. 61), using zinc dust to prevent bumping, receiving the distillate into standard acid, and titrating against standard alkali.

Nitrogen Determination in the Ether Extract.—Ten grams of the sample are extracted with absolute ether for twenty hours in a continuous-extraction apparatus, the extract being collected in a tared Kjeldahl extraction- and distillation-flask, the same as used in the preceding section. The ether is then evaporated off, the residue dried to constant weight at 110° C. and its weight ascertained. The nitrogen is then determined in the ether extract by the Gunning-Arnold method.

Determination of Piperin.†—Fifty grams of the sample are thoroughly exhausted with hot alcohol, and the alcohol extract evaporated to dryness. The dry residue is then treated with a solution of potassium hydroxide, and washed upon a filter. The residue is washed several times with the caustic alkali, which dissolves the resinous matters, and afterwards with water. It is then dissolved in alcohol, from which crystals of crude piperin separate on evaporation. These are redissolved in alcohol, and precipitated by the addition of water. The crystalline precipitate is collected on a tared filter, washed with water, dried, and weighed.

Piperin may be roughly estimated by multiplying the nitrogen in the ether extract by the factor 20.36.

The amount of piperin varies considerably, ranging in black pepper from 4 to 9 per cent.

Microscopical Characteristics of Ground Pepper.—Moeller's representation of powdered black pepper shows what should be looked for under the microscope with the best conditions (Fig. 78). The shell of the peppercorn, a cross-section of which is shown at (1), consists of the

* Forty grams K_2S in 1 liter of water.

† Villiers et Collin, Substances Alimentaires, p. 371.

epidermis, *a*, under which is a thin layer of brown parenchyma, *c*, while below this layer is shown the most characteristic portion of the pepper shell, viz.: the thickened, colored, stone cells, *b*. These are as a rule inclined to be rectangular rather than rounded. At *d* is shown a bit of the colorless parenchyma of the fruit itself.

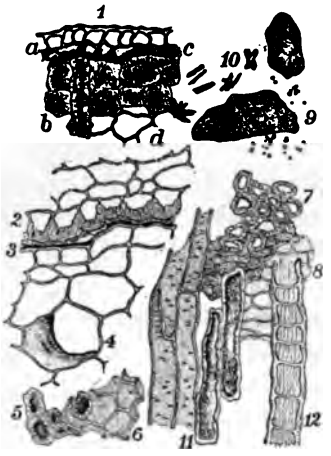


FIG. 78.—Powdered Black Pepper under the Microscope. × 125. (After Moeller.)

(2), (3), and (4) show a cross-section of the outer part of the berry, (2) representing the inner stone-cell layer, a single row of horseshoe-like cells, (3) the thin seed coat, and (4) the white perisperm, with its large cells. Here and there through the perisperm certain yellow contents are visible, consisting largely of resinous matter. A dark resin cell is shown at (4). The ethereal oil, starch, and piperin are found in this part of the berry.

(5) shows in plan view the mostly rectangular stone cells of the pepper shell, resting upon the epidermis (6). Groups of stone cells are frequently thus found with portions of the epidermis.

The inner rounded, or cup-shaped cells are shown in plan view at (7) and the seed skin at (8), masses of starch and separate starch granules are shown at (9), and crystals of piperin at (10).

The bast-parenchyma of the pepper stem is shown at (11), pieces of which are commonly found in powdered pepper, and (12)

by no means as clearly as by the use of chloral hydrate. Large polygonal masses of starch appear grouped as photographed in Fig. 256, Pl. XXXIV, if not rubbed out too fine under the cover-glass. Starch, indeed, is the most conspicuous element of pepper, being distributed more or less evenly throughout the mass. The powder may, however, be so finely reduced by abrasion under the cover-glass as to break up these starch masses wholly or in part, so that the granules may appear in much smaller groups or even singly. Fig. 255 shows such a field under a higher magnification. The individual granules of pepper starch average 0.003 mm. in diameter.

Besides the starch, and next to it the most numerous, one finds in the water-mounted black-pepper specimen many of the dark-yellow, thick-walled stone cells, patches of the colored parenchyma, and epidermis of the shell. Other elements of the perisperm, besides the starch, are seen in fragments, such as bits of resin, small droplets of oil, pieces of stems, and occasionally the needle-shaped crystals of piperin. Some of the rounded, cup-shaped cells are also usually found.

White pepper contains, of course, the same elements, but without the deeply colored stone cells and other characteristics of the shell, which has been removed from it.

Adulteration of Pepper.—The following U. S. standards for pepper have been adopted: For white pepper, non-volatile ether extract should not be less than 6%; starch should not be less than 53% by the diastase method, nor less than 56% by direct inversion; total ash should not be more than 4%; ash insoluble in hydrochloric acid should not exceed 0.5%; crude fiber should not exceed 5%. One hundred parts of the non-volatile ether extract should contain not less than 4 parts of nitrogen. For black pepper, which should be free from added pepper shells, pepper dust, and other pepper by-products, non-volatile ether extract should not be less than 6%; starch by the diastase method should not be less than 22%, and by direct inversion should not be less than 28%; total ash should not exceed 7%; and crude fiber should not exceed 15%. One hundred parts of the non-volatile ether extract should contain not less than 3.25 parts of nitrogen. The adulterants used in ground pepper are many and varied.

Pepper Shells, which have been removed from the white pepper of commerce, are not infrequently ground and added to the cheaper grades of black pepper. When a sample of black pepper is shown by the microscope to contain more shells in proportion to the other elements than could be possible in a ground whole berry, added shells are indicated.

The analyst should, for comparison, grind in a mortar single berries of various grades, and familiarize himself with the appearance of the ground powder under the microscope, when the maximum amount of shells possible under natural conditions are present, noting especially the apparent number of stone cells of the outer coating. The familiar title of P. D. (pepper dust) originally given to ground pepper shells, stems, and "sweepings" is now applied in the trade not only to almost any cheap and appropriate material for admixture with pepper, but also, in a broader sense, to ground powder suitable as an adulterant for any spice.

Ground Olive-stones constitute one of the most commonly found foreign materials used as an adulterant of pepper. The powder, sometimes called "poivrette," is very like white pepper in appearance, is wholly inert in taste, and thus forms an admirable adulterant. While best detected by their characteristic appearance under the microscope, the presence of ground olive stones may be shown by color tests with certain chemical reagents.

Pabst has adopted for this purpose a test first suggested by Wurster for the detection of wood pulp in paper. The reagent is prepared as follows: In a porcelain capsule 10 grams of commercial dimethyl anilin are mixed with 20 grams of pure concentrated hydrochloric acid, and at least 100 grams of cracked ice are added. Then, while stirring, a solution of 8 grams of nitrite of soda in 100 cc. of water are added little by little, and the mixture allowed to remain for half an hour, after which 30 or 40 cc. of hydrochloric acid are added, and 20 grams of tin-foil. The reduction is allowed to go on for half an hour, heating on the water-bath, if necessary. The tin is then precipitated by granulated zinc, the liquid is filtered, and the filtrate neutralized with carbonate of potassium or sodium to the point of forming a precipitate, the precipitate being



Jumeau uses for a color reagent 5 grams of iodine in 100 cc. of a mixture of equal parts of ether and alcohol. Enough of the finely ground pepper to be examined is placed in a porcelain capsule to cover the bottom of the dish, and sufficient iodine reagent is added to wet the entire mass, carefully avoiding excess. The thick paste is first mixed till homogeneous, and then allowed to dry in the air, after which it is broken up by a pestle, and the powder examined, either under the microscope, or by the naked eye. With pure pepper, a more or less deep-brown color is produced uniformly through the powder, but if olive stones are present, particles of these are colored yellow. With the naked eye as small an admixture as 2% of olive stones can thus be detected.

A solution of anilin acetate colors olive stones yellowish brown, while pure pepper appears grayish, or white.

Under the microscope olive stones are readily apparent, since the stone cells differ in size, form, and mode of grouping from those of pepper. Fig. 263, Pl. XXXVI, is a photograph of a water-mounted specimen of olive stones. They are for the most part entirely devoid of color, being long and narrow. In shape and manner of grouping they much resemble cocoanut shells (p. 321), but are distinguished from the latter from their lack of color.

Fig. 261 shows under low magnification a sample of pepper, bought on the market in Massachusetts, highly adulterated with olive stones. A large mass of the stone cells of the adulterant appears in the center of the field. Many of the stone cells are shown arranged end to end, so that what at first sight appear to be single, very long cells are in reality made up of several shorter ones. In ground olive stones one frequently finds, besides the stone cells, bits of the outer tegument of the seed, showing large cells with sinuous, rather thick walls; also bits of parenchyma, crossed frequently by fibro-vascular duct bundles.

Buckwheat Middlings constitute a material much used as an adulterant of pepper. The starch of buckwheat possesses the added advantage, from the point of view of the spice-grinder, that it somewhat resembles pepper starch in microscopical appearance, not only in the shape of the starch granules, but also in the manner of grouping into masses. Compare Figs. 128 and 129, Plates II and III, showing buckwheat starch, with Figs. 255 and 256, Pl. XXXIV, respectively, showing pepper starch made under similar conditions of magnification, etc. The two are readily distinguished by difference in size. The starch granules and masses are coarser in the case of buckwheat than of pepper.

Fig. 260, Pl. XXXV, shows a photograph of a pepper sample adulterated with buckwheat, masses of both starches appearing in the same field.

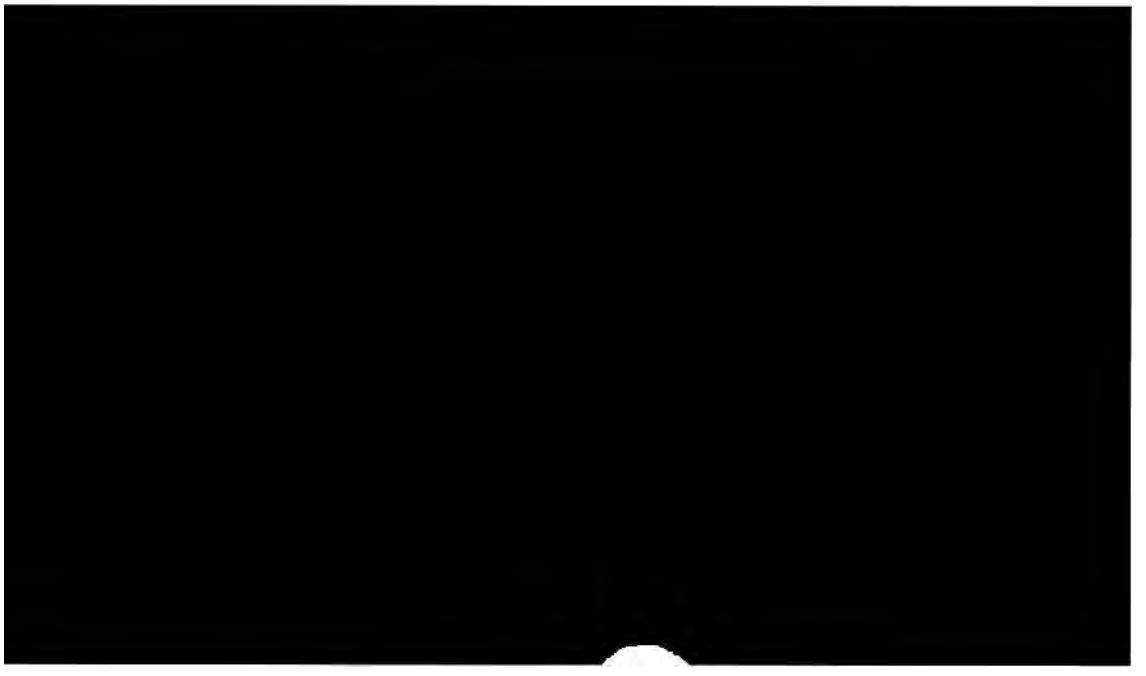
Other Adulterants found in Massachusetts samples of pepper have been wheat and corn products, nutshells, cayenne, charcoal, turmeric, rice, sand, and sawdust.

Long Pepper, according to English analysts, has been used to a considerable extent as an adulterant. This is the fruit of the *Chavica Roxburghii*, a wild plant growing in India on the banks of rivers. The fruit, as its name implies, is long and cylindrical, while of about the same diameter as the spherical true peppercorns. It is undoubtedly a form of pepper, in that the fruit contains the same ingredients as true pepper, viz.: starch, resin, piperin, etc. Long pepper contains, as a rule, less than half the amount of piperin that true pepper does, and rather more starch than black pepper. Its taste is much less pungent than that of true pepper.

From its method of growth, long pepper is found with considerable dirt and sand adhering to the outer surface of the dried grains. This is due to the fact that the fruit often trails on the ground, and in gathering it the natives are not particular about removing the adhering soil. The surface of the fruit grains being very rough and irregular, much of the dirt remains dried thereon. The presence of long pepper thus materially increases the ash.

Long pepper possesses a very disagreeable, but peculiar odor, developed more especially when slightly warmed. For this reason, if for no other, it is not an ideal adulterant, since pepper containing it would not be palatable with warm food.

Brown gives the following analyses of samples of long pepper:



of starch granules and his experience is shared by the writer. When the two specimens (long and true pepper) are viewed side by side in water mounts under the microscope, the average size of the long pepper-starch grains is a trifle larger than those of true pepper, though, unless compared directly, the difference is not readily apparent. Stokes suggests a method of distinguishing the two by polarized light. With crossed Nicols, so that a dark field is given, and with the specimen mounted in glycerin, true pepper starch shows an evenly dark appearance, using a low power, while with long pepper a "ghostly white" image is shown. Long pepper, when present in true pepper powder, may generally be rendered apparent by the development of the characteristic odor on heating. It may also be detected by spreading out particles of the powdered material on paper, and examining them with a magnifying-glass. Bits of fluffy fiber from the catkin of the long pepper will always be found in the ground powder, and will be apparent under the magnifying-glass.

CAYENNE.

Nature and Composition. — Cayenne pepper of commerce is the ground dried fruit pods of several species of *Capsicum*, a genus of the nightshade family (*Solanaceæ*), indigenous to the American tropics, but now cultivated in nearly all warm countries. The species furnishing the most common sources of Cayenne pepper are *Capsicum annuum*, *Capsicum frutescens*, *Capsicum fastigiatum*, *Capsicum longum*, and *Capsicum baccatum*. The kitchen garden variety of ordinary red pepper is for the most part the *Capsicum annuum*, of which there are over thirty varieties cultivated in the United States. The Cayenne and Chili varieties are most highly prized, because of their pungency. The fruits of these are somewhat long and slender. Other varieties are of larger size and milder.

Paprika or Hungarian red pepper is a very mild variety of *Capsicum annuum* much prized on account of the intense red color of its pods. The powder is of a much deeper color than that of ordinary varieties of cayenne.

The capsicum plant has solitary flowers, with a five-cleft corolla, and the fruit is of an elongated, conical form. The surface of the fresh fruit is smooth and very red, but it loses some of its brilliance in drying, and becomes shriveled. The pericarp is thin and tough, and at its base is a five-lobed calyx, greenish brown in color, terminating in a thick stem.

The fruit proper is divided into two or three cells, which are separate and distinct at the lower portion, but which unite and form one at the top. The cells inclose a large number of yellow, wrinkled, kidney-shaped seeds, containing a fleshy albumen, and a curved embryo.

Cayenne contains a fixed, bland oil, found in both pod and seed, but more abundantly in the latter, considerable resinous and mucilaginous material, a red coloring matter confined to the pod, and the active principle *capsicin*, a crystalline alkaloid, to which much of the pungency is due. The capsicin is present in both seeds and pod, but is more abundant in the latter, where it is dissolved in the oil.

Capsicin may be isolated, according to Thresh, by extracting powdered cayenne with petroleum ether, mixing the red residue left on evaporating off the solvent with two or three times its weight of oil of almonds, and exhausting the mixture with alcohol. On evaporating the alcohol extract, the capsicin crystallizes out in narrow, thin plates, very soluble in alcohol, but insoluble in water. They volatilize at 100°, and condense in small drops.

The red coloring matter is soluble in ether, petroleum ether, carbon bisulphide, and chloroform, but sparingly soluble in alcohol.

Richardson * gives the following data of analyses of two pure samples of cayenne:

	Water.	Ash.	Fixed Oil.	Volatile Camphor, etc.	Fiber.	Albuminoids.	Undetermined.	Total.	Nitrogen.
A.....	2.35	9.06	0.12	26.99	16.88	13.13	41.47	100	2.10
B.....	5.74	5.24	1.58	17.90	18.10	11.20	40.24	100	1.70

Winton, Ogden, and Mitchell analyzed eight samples, representing three varieties of cayenne, with the following summarized results:

	Moisture.	Ash.			Ether Extract.	
		Total	Soluble in Water.	Insoluble in HCl.	Volatile.	Non-volatile.
Maximum	7.08	5.96	4.93	0.23	2.57	21.81
Minimum	3.67	5.08	3.30	0.05	0.73	17.17
Average	5.73	5.43	3.98	0.15	1.35	20.15

	Alcohol Extract.	Reducing Matters as Starch, Acid Conversion.	Starch by Diastase Method.	Crude Fiber.	Nitrogen, X 6.25.	Total Nitrogen.
Maximum	27.61	9.31	1.46	24.91	14.63	2.34
Minimum	21.52	7.15	0.80	20.35	13.31	2.13
Average	24.35	8.47	1.01	22.35	13.67	2.18

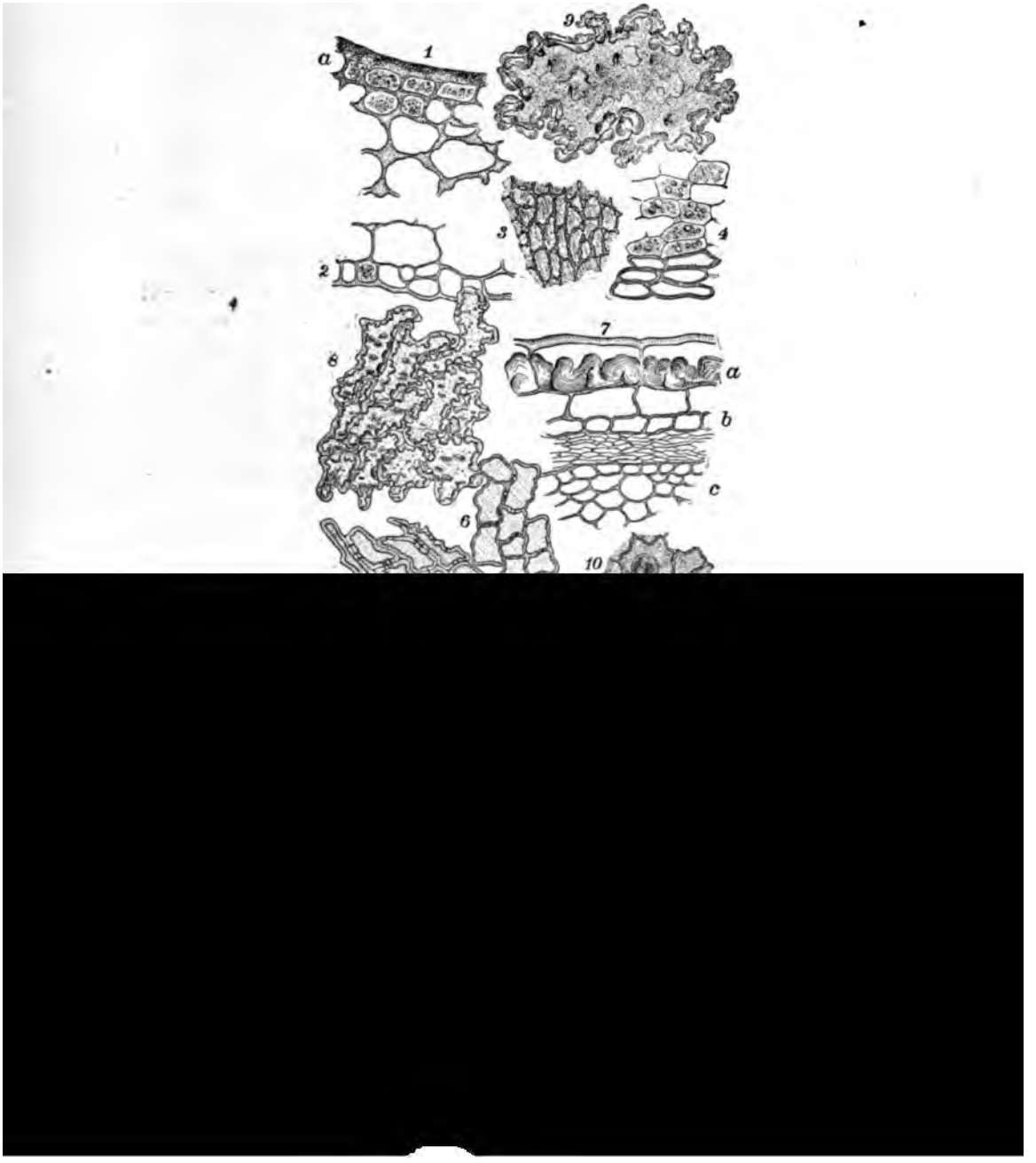
Microscopical Structure of Cayenne.—Fig. 79, from Moeller, shows the appearance under the microscope of various elements of powdered cayenne. (1) is a sectional view through the outer portion of the fruit shell or pod, showing the epidermis *a*, and beneath this the collenchyma layer *b*. The inner epidermis is shown at (2), with its cells thick-walled in places, and inclosing brilliant, red oil drops of coloring matter. (3) represents the outer and (4) and (5) the inner epidermis in plan view. In (5) the oil drops are more clearly shown. The outer epidermis of the variety known as Chillies is shown at (6).

A cross-section through the seed shell is shown at (7), *a* being the epidermis of the seed, *b* the parenchyma layer directly beneath, and *c* the albumen of the endosperm. (8) shows in plan view the peculiar seed epidermis, the appearance of which Moeller compares with that of tripe. At (9) is shown one of the isolated cells of the seed epidermis more highly magnified, while (10) shows the epidermis of the calyx.

Figs. 211 and 212, Pl. XXIII, show photomicrographs of powdered cayenne. In Fig. 211 is shown a large bit of the outer epidermis of the fruit pod, while in Fig. 212 appears a smaller portion of this same kind of epidermis, and next to this the tripe-like skin of the seed shell, with its striking markings suggestive of the convolutions of the intestines. Yellow or yellowish-red droplets of oily coloring matter are distributed through the field. Cayenne has no starch.

Adulteration of Cayenne.—The U. S. standards for cayenne are the following: Non-volatile ether extract should be not less than 15%; total ash should not exceed 6.5%; ash insoluble in hydrochloric acid should not exceed 0.5%; starch by the diastase method should not exceed 1.5%, and crude fiber should not exceed 28%.

The most common adulterants of cayenne are the starches of the cereal grains, corn and wheat. Ground pilot bread and crackers are especially common. Besides these the writer has found in the routine



An abnormally high ash is suggestive of adulteration. According to Vedrodi, the ash of genuine cayenne should not exceed 5.96. The presence of red ocher is rendered apparent by the high content of iron.

Salts of lead and mercury are rarely if ever now used for color.

Ground Redwood.—Numerous varieties of redwood are commonly used to intensify the color of cayenne, especially when otherwise highly adulterated with colorless materials, such as the starches. The redwood is sometimes used alone, and sometimes in mixture with turmeric. Both redwood and turmeric are readily recognized under the microscope.

Fig. 214, Pl. XXIV, shows a cayenne sample adulterated with corn starch and red sandalwood, a mass of the latter filling the center of the field. The wood fibers of the dyestuff, even when finely ground, are very striking under the microscope, showing a brick-red color.

Detection of Coal-tar and Vegetable Colors.—Oil-soluble coal-tar dyes and vegetable colors may be tested for in cayenne by an adaptation of Martin's butter-color method, shaking the ether extract of the sample with the alcohol and carbon bisulphide mixture, page 434. The carbon bisulphide dissolves the oil and natural color of the cayenne, while the overlying alcohol layer would hold in solution many of the artificial coloring matters that may be employed.

The natural color of cayenne is sparingly soluble in alcohol, but readily soluble in carbon bisulphide. The separated alcohol is examined for colors by methods given elsewhere.

Szigeti* treats the suspected sample of cayenne with water acidified with acetic acid, and boils in this solution a bit of wool, which, if carotin or a coal-tar dye be present, is colored red. If the color is carotin, it will be removed from the wool by treatment with petroleum ether, or by heating at 100° C. for some hours, but if a coal-tar dye, it will still remain fixed thereon.

GINGER.

Nature and Composition.—Ginger as a spice is the ground root-stock of the *Zingiber officinale*, an annual herb of the family of *Zingiberaceæ*, growing to a height of from 3 to 4 feet. It is a native of India and China, but is cultivated quite extensively in tropical America, Africa, and Australia.

The root is dug when the plant is a year old, and when the stem has

* Zeits. landw. Versuchs. Österreich, 1902, 5. 1208, 1222.

withered. If the root, when freshly dug and scalded to prevent sprouting, is dried at once, it forms the so-called black ginger. When decorticated, it furnishes what is known in commerce as white ginger. The best variety of the latter is Jamaica ginger. The scraped root is sometimes bleached to make it still whiter, or sprinkled with carbonate of lime.

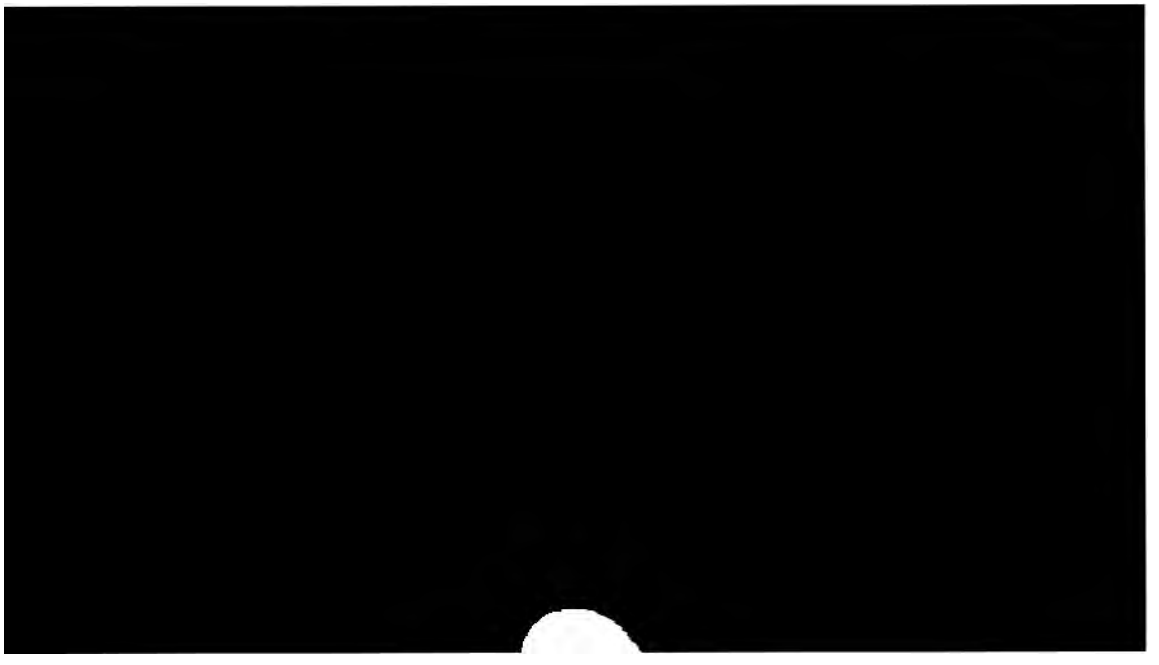
In commerce whole or black ginger appears in fragments 4 to 10 cm. long, and from 10 to 15 mm. in diameter. These usually have three or four various-sized, irregular branches, some short and thick, others elongated. The epidermis is gray or yellowish gray in color, more or less wrinkled, and beneath it is a reddish-brown layer. The inner portion of the dried root is white or yellowish. The root is hard, and of a compact, horny structure.

White or decorticated ginger appears in longer fragments, but of smaller diameter than the others, is less aromatic, and as a rule less highly prized. The principal part of the ginger of commerce comes from Calcutta. Preserved ginger root is prepared by boiling the root in water, and curing with sugar. Much of the preserved ginger comes from Canton.

The distinguishing features of ginger are its large content of starch, its volatile oil, and its resinous matter. Inasmuch as the epidermis contains a large amount of pungent resin, it is easy to see how the peeled or decorticated variety is inferior.

Oil of ginger is very aromatic, and of a greenish-yellow color. Its specific gravity ranges from 0.875 to 0.885. It is slightly soluble in alcohol. Of its composition little is known.

Richardson's analyses in full of five samples of whole ginger-root are as follows:



	Moisture.	Ash.			Lime (CaO).	Ether Extract.	
		Total.	Soluble in Water.	Insoluble in HCl.		Volatile.	Non-volatile.
Ginger: Maximum	11.72	9.35	4.09	2.29	3.53	3.09	5.42
Minimum	8.71	3.61	1.73	0.02	0.20	0.96	2.82
Average	10.44	5.27	2.71	0.44	0.80	1.97	4.10
Exhausted ginger from English ginger-ale works	10.61	2.12	0.59	0.18	.....	1.61	3.86
Exhausted ginger from extract works	8.02	5.05	3.55	1.50	.....	0.13	0.54

	Alcohol Extract.	Reducing Matters by Acid Conversion, as Starch.	Starch by Diastase Method.	Crude Fiber.	Nitrogen, $\times 6.25$.	Cold water Extract.	Total Nitrogen.
Ginger: Maximum	6.58	62.42	60.31	5.50	9.75	17.55	1.55
Minimum	3.63	53.43	49.05	2.37	4.81	10.92	0.77
Average	5.18	57.45	54.53	3.91	7.74	13.42	1.23
Exhausted ginger from English ginger-ale works	4.88	59.86	54.57	5.17	6.94	6.15	1.11
Exhausted ginger from extract works	1.52	.....	.....	.....	.....	16.42	

McGill * records the analyses of ninety-eight samples of ground ginger as sold in the Canadian market. Of thirty-two of these, pronounced pure on analyses, the following is a summary:

	Moisture or Loss on Drying at 100°.	Petroleum-ether Extract.	Cold-water Extract.	Ash.			
				Total.	Soluble.	Insoluble.	Alkalinity of Soluble Ash as K_2O.
Maximum	12.00	6.13	15.48	7.84	3.15	3.99	.133
Minimum	9.50	2.78	14.04	3.67	2.28	1.96	.103

According to Vogl, the proportion of ginger ash varies quite widely according to the kind, but should never exceed 8%.

Exhausted Ginger and Methods of Detection.—There are two kinds of exhausted ginger commercially available for admixture with ground spice, as an adulterant. One is the product left after extraction with strong alcohol in the making of extract of Jamaica ginger, and the other the residue from extraction with either very dilute alcohol, or with water,

* Dept. Inl. Rev. Canada Bul. 48, pp. 10, 11.

in the manufacture of ginger ale. Ground, exhausted ginger is rarely substituted wholly for the pure variety, since, from its lack of pungency, the sophistication would be too apparent. It is rather used to mix with the latter in varying proportions, and as an adulterant of other spices.

Ginger that has been exhausted by extraction with alcohol has been deprived of most of its volatile oil, which is found in the "extract," while for the manufacture of ginger ale, a water extract, or at most a very dilute alcoholic extract is best adapted. Such a water extract does, as a matter of fact, remove much of the valued pungency, so that the residue, or exhausted ginger, is rather inert.

Either the alcohol- or the water-extracted variety of exhausted ginger, when present in considerable amount, would be apparent, one by the alcohol and ether extract, and the other by the abnormally low cold-water extract, and water-soluble ash.

Dyer and Gilbard * first called attention to the water-soluble ash as a reliable means of indicating exhausted ginger. Six samples of ginger of known purity were analyzed by them, their results being summarized as follows:

		Total Ash.	Water-soluble Ash.	Alcoholic Extract after Ether Extract.
Pure ginger (6 samples):	Highest	4.1	3.	3.8
	Lowest	3.1	1.9	2.1
	Average	3.8	2.7	2.8
Exhausted ginger (6 samples):	Highest	2.3	0.5	1.5
	Lowest	1.1	0.2	0.8
	Average	1.8	0.35	1.2



stand at rest for sixteen hours in addition. The contents are then filtered, and 50 cc. of the filtrate evaporated to dryness in a platinum dish. It is then dried at 100° to constant weight and weighed.

Microscopical Structure of Ground Ginger.—Fig. 80, from Moeller, shows elements of ginger root, from which the epidermis has not been

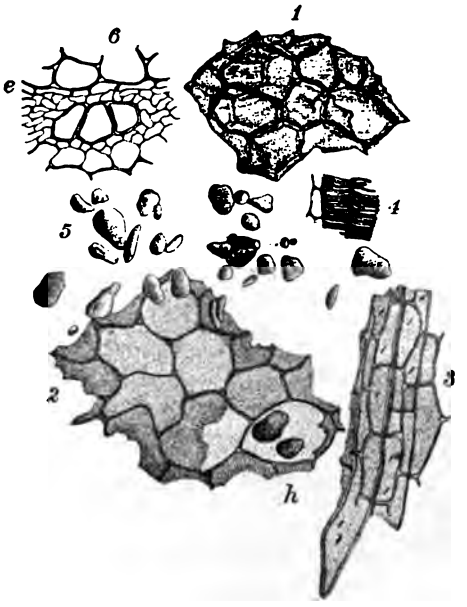


FIG. 80.—Powdered Ginger under the Microscope. ×125. (After Moeller.)

removed. A bit of the large-celled cork (or dead protective tissue of the epidermis) is shown in plan view at (1); at (2) is shown in cross-section the parenchyma in which the starch is contained, *h* being an oil-cell; (3) shows the parenchyma in longitudinal section, with bast fibers. Fragments of spiral ducts are shown at (4), and starch grains at (5). (6) is a cross-section in the extreme interior of the root.

The most prominent feature of powdered ginger is the starch grains (5), which Moeller compares in shape to tied sacks.

Fig. 228, Pl. XXVII, is a photomicrograph of pure, ground ginger, mounted in water, showing the starch grains inclosed in the cells of the parenchyma. Fig. 231 shows the starch grains alone. The granules of ginger starch are ellipsoidal, and as a rule very clear and transparent, being for the most part entirely devoid of either hila or concentric rings.

Occasionally granules are to be found, however, with faint concentric markings, and even with an apparent hilum. The characteristic form of the ginger starch granule is more or less egg-shaped, with a small protuberance near one end. This protuberance serves to readily distinguish the starch granules of ginger from those of wheat, with which ginger is frequently adulterated. While wheat granules are of various sizes, the grains of ginger starch are as a rule much more uniform.

Adulteration of Ginger.—U. S. standard ginger should meet the following requirements: Starch by the diastase method should not be less than 42%, and by the acid-conversion method should not be less than 46%; crude fiber should not exceed 8%; total ash should not exceed 8%; lime should not exceed 1%; ash insoluble in hydrochloric acid should not exceed 3%.

Besides exhausted ginger, the most common adulterants found in powdered ginger are turmeric, wheat, corn, rice, and sawdust. Sawdust of soft wood is a not uncommon adulterant, and care should be taken to distinguish between the wood fiber natural to the ginger root, and that of the foreign variety. A careful study should be made of finely ground, soft-wood sawdust, with its long spindle cells and lateral pores, as shown in Fig. 266, Pl. XXXVII, and the wood fiber of the genuine ginger root. A large admixture of sawdust would materially increase the percentage of crude fiber.

Fig. 234, Pl. XXIX, shows a sample of ginger adulterated with corn and wheat. Fig. 232 shows a mass of wheat bran in an adulterated sample.

Fig. 233 shows ginger adulterated with turmeric.*

TURMERIC.



Curcumin ($C_{14}H_{14}O_4$) is insoluble in cold water, but readily soluble in alcohol. It is extracted from powdered turmeric by boiling the latter with water, filtering, and extracting the residue with boiling alcohol. The alcoholic solution is filtered, evaporated, and the residue extracted with ether. The ether extract contains the curcumin, together with a small amount of volatile oil.

Curcuma oil is an orange-yellow, slightly fluorescent liquid, its specific gravity being 0.942.

The following analyses of turmeric were made in the writer's laboratory:

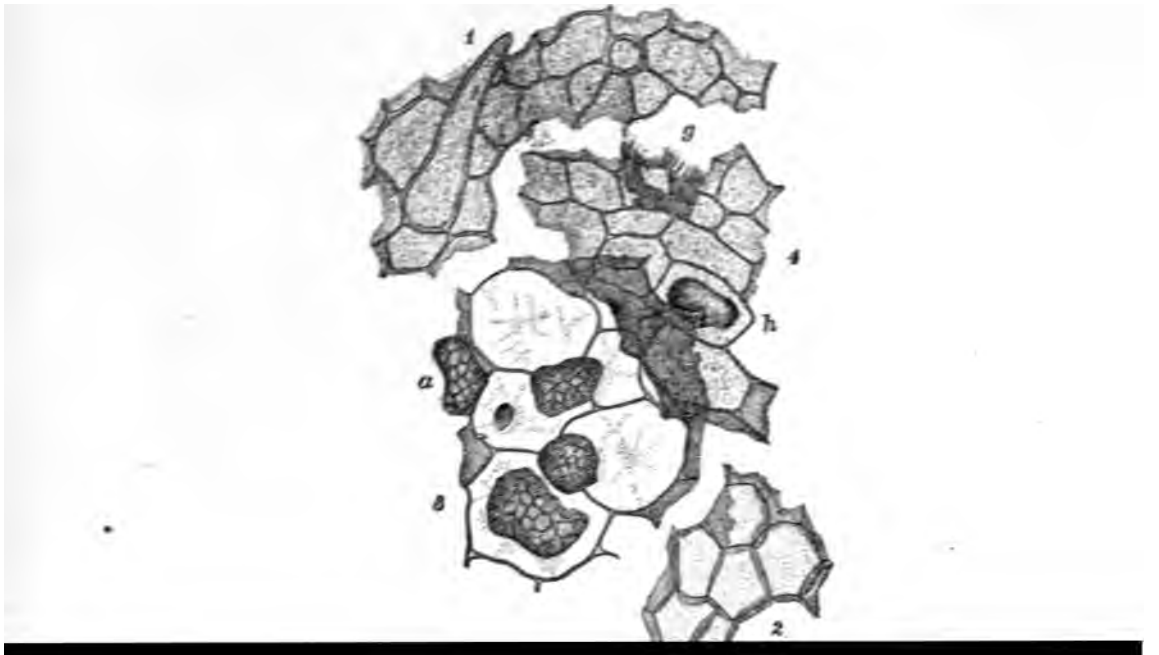
Variety.	Moisture.	Total Ash.	Ash Soluble in Water.	Ash Insoluble in HCl.	Total Nitrogen.	Protein. N × 6.25.	Total Ether Extract.
China	9.03	6.72	5.20	0.11	1.73	10.81	10.86
Pubna	9.08	8.52	6.14	....	0.97	6.06	12.01
Alleppi	8.07	5.99	4.74	....	1.56	9.75	10.66
Average	8.73	7.07	5.36	....	1.42	8.88	11.17

Variety.	Volatile Ether Extract.	Non-volatile Ether Extract.	Alcohol Extract.	Crude Fiber.	Reducing Matter by Acid Conversion, as Starch.	Starch by Diastase Method.
China	2.01	8.84	9.22	4.45	48.69	40.05
Pubna	4.42	7.60	7.28	5.84	50.08	29.56
Alleppi	3.16	7.51	4.37	5.83	50.44	33.03
Average	3.19	7.98	6.96	5.37	49.73	34.21

Microscopical Structure of Turmeric.— Moeller's representation of characteristics of powdered turmeric is reproduced in Fig. 81. The epidermis is shown at (1) with one of the numerous, one-celled hairs that grow from it, also the scar left after one of the hairs has been removed; (2) shows in plan view the cork immediately under the epidermis. The tender-celled parenchyma is shown in cross-section at (3), and in longitudinal section at (4). In some of the cells of the parenchyma are found dark-yellow lumps of resin (*h*), and vascular ducts (*g*), but by far the most numerous and striking contents of the parenchyma-cells are the bright-yellow masses of "paste balls" (3*a*) and the starch granules, one of which is shown in (3). See also Plate XIII. The starch grains in the water-mounted powder show under the microscope in masses, usually of a deep-yellow color, unless very finely rubbed out, when they appear for the most part in fragments.

The whole starch granule appears somewhat in the form of a clam-shell, with very distinct markings. When fragments of the starch granules are carefully examined, these distinct markings are so strongly characteristic, even in the smallest pieces commonly found in the powdered sample, as to nearly always serve to identify them. See Fig. 171, Pl. XIII.

Turmeric as an Adulterant.—Turmeric is a material especially adapted by its deep-yellow color to intensify mustard and ginger, especially when



Turmeric, when present to any marked extent in a powdered spice, may be detected chemically, by extracting the material with alcohol, pouring off the latter, and soaking in it a piece of filter-paper. Turmeric, if present, will stain the latter yellow, turning red with alkali, especially apparent after drying. Soak the yellow paper in a solution of borax, acidulated slightly with hydrochloric acid. When dry, a rose-red color will indicate turmeric, turning dark olive when dilute alkali is applied.

MUSTARD.

Nature and Composition.—Mustard is the seed of the mustard plant, an annual belonging to the family *Cruciferæ*, and to the genus *Sinapis*, or *Brassica*, as it is sometimes called. The plant is an herb, native throughout Europe, and cultivated extensively in the United States. It grows to a height of from 3 to 6 feet, having yellow flowers and lyrate leaves.

Two varieties commonly used are *Brassica* or (*Sinapis*) *alba*, white mustard, and *Brassica* (or *Sinapis*) *nigra*, black mustard, the ground spice being as a rule a mixture of the two. In the trade these varieties are known as brown and yellow mustard respectively. The seeds of both varieties are globular, those of the black mustard being small, and of a dark-brown color on the outside and yellow within. White mustard seeds are considerably larger than the black, being pale yellow in color on the outside.

The surface of the black mustard seeds is reticular, and full of small depressions, while the white variety is much smoother. There are several layers forming the husk of the seed of both varieties, and within the husk is the yellowish-colored kernel or embryo, with two cotyledons.

Both black and white mustard contain from 31 to 37% of fixed oil, a soluble ferment known as myrosin, and a sulphocyanate of sinapin. Mustard seeds contain no starch, and very little volatile oil as such. Black mustard seed contains sinigrin, or myronate of potash (not found in the white seed), which, when moistened with water, forms by hydrolysis the volatile oil of black mustard, otherwise known as allyl isothiocyanate, in accordance with the following equation:

$$\underset{\text{Potassium myronate}}{KC_{10}H_{18}NS_2O_9} + H_2O = \underset{\text{Glucose}}{C_6H_{12}O_6} + \underset{\text{Mustard oil}}{C_3H_5CNS} + \underset{\text{Potassium bisulphate}}{KHSO_4}.$$

Mustard Oil (volatile) is a colorless, or slightly yellow, highly refractive liquid of a very strong odor, and capable of blistering the skin when

brought in contact with it. It is optically inactive. Its specific gravity varies between 1.016 and 1.030. It boils between 148° and 156°. It turns reddish brown by exposure to light.

Volatile oil of black mustard forms thiosinamine with ammonia, as follows:

$$C_3H_5CNS + NH_3 = CS.NH_2.NH.C_3H_5.$$

Thiosinamine is soluble in hot water, from which it crystallizes in tufts of monoclinic crystals, having a melting-point of 74° C. It is precipitated by silver nitrate, mercuric chloride, and Mayer's solution.

White mustard differs from the black in containing a sulphur compound, *sinalbin*, $C_{30}H_{42}N_2S_2O_{15}$. This is a glucoside. Sinalbin by hydrolysis forms an oil of white mustard, in a somewhat similar manner to the potassium myronate of black mustard, and according to the following equation:

$$\underset{\text{Sinalbin}}{C_{30}H_{42}N_2S_2O_{15}} + H_2O = \underset{\substack{\text{Sinalbin}\\\text{mustard oil}}}{C_7H_7ONCS} + \underset{\text{Glucose}}{C_6H_{12}O_6} + \underset{\substack{\text{Sinapin acid}\\\text{sulphate}}}{C_{16}H_{24}NO_5HSO_4}.$$

Sinalbin Mustard Oil cannot be obtained by the distillation of white mustard, being sparingly volatile with steam.

Sinalbin mustard oil somewhat resembles that from black mustard, being quite as pungent, but less strong in odor when cold. It is soluble in dilute alkali.

Fixed oil of mustard is a bland, tasteless, and nearly odorless oil, its specific gravity at 15° varying between the limits of 0.914 to 0.918. It is said to be used to some extent as an adulterant of table oils, being separated by pressure from the crushed mustard seeds before the latter are ground into "flour." The chief use of mustard oil is in mixture

mustard is molded together into thin, hard plates, called "mustard cake." This is then broken up and reduced to fine powder by pounding.

Richardson's* analyses of whole-seed flour, prepared by himself without the removal of the fixed oil, are as follows:

	Water.	Ash.	Volatile Oil.	Fixed Oil.	Starch.	Crude Fiber.	Albuminoids.	Undetermined.	Nitrogen.
White seed	5.57	4.29	.97	33.56	.00	5.40	28.88	21.33	4.62
White flour	3.33	5.23	1.84	34.83	.00	9.05	25.56	20.16	4.09
Seed husk	6.17	4.99	.55	28.12	.00	9.50	23.44	27.23	3.75
California yellow	4.83	5.96	1.27	31.96	.00	8.50	31.13	16.35	4.98
California brown	4.11	4.88	1.35	36.63	.00	16.18	24.69	12.16	3.95
English yellow	3.11	4.07	2.06	31.51	.00	6.90	30.25	22.10	4.84
Trieste brown	4.62	5.61	.63	39.55	.00	10.84	25.88	18.87	4.14

Winton and Mitchell made no full analyses of mustard seed of known purity, but the following is a summary of analyses of 18 samples of commercial mustards, sold in packages in Connecticut, and not found to be adulterated:

	Total Ash.	Ether Extract.		Reducing Matters by Acid Conversion, as Starch.	Starch by Diastase Method.	Crude Fiber.	Nitrogen ×6.25.
		Volatile.	Non-volatile.				
Maximum	7.35	1.90	28.10	6.12	2.08	4.87	43.56
Minimum	4.81	0.00	17.14	1.85	0.28	1.58	35.63
Average	5.99	0.56	20.61	4.33	1.07	2.58	39.57

The following analyses of 5 samples of mustard flour, 6 samples of mustard hulls, and 6 samples of whole mustard, were made in the author's laboratory in 1903:

* U. S. Dept. of Agric., Div. of Chem., Bul. 13, part 2.

	Water-soluble Ash.	Ash Insoluble in HCl.	Total Ether Extract.	Volatile Ether Extract.	Non-volatile Ether Extract.	Alcohol Extract.	Total Nitrogen.	Volatile Oil.	Crude Fiber.	Reducing Matters by Acid Conversion.	Reducing Matters by Diastase.	On Dry Fat-free Substance. Alcohol Extract.	Total Nitrogen.	Total Ash.	Crude Fiber.	Reduc'g Matters by Acid Conversion.	Reduc'g Matters by Diastase.
	.27	.08	17.46	0.0	17.46	25.31	6.37	3.98	3.28	11.89	.71	32.88	8.27	7.35	4.26	15.43	.93
	.23	.13	20.64	0.0	20.64	19.22	6.75	4.67	2.27	4.87	.23	26.64	9.36	6.79	3.15	6.75	.32
	.09	.50	16.28	0.0	16.28	21.98	7.13	3.09	2.45	5.63	.22	29.61	9.61	7.07	3.30	7.59	.30
	.19	.24	18.10	0.0	18.10	22.17	6.75	3.91	2.67	7.47	.39	29.71	9.08	7.04	3.57	9.02	.52
	.18	.35	12.65	0.0	12.65	24.21	7.44	.....	1.87	6.12	.23	29.03	9.20	5.85	2.31	7.57	.28
	.22	.30	25.95	0.0	25.95	20.78	6.21	.....	2.21	5.75	.00	30.13	9.00	6.81	3.20	8.34	.00
	.20	.32	19.30	0.0	19.30	22.49	6.83	.....	2.04	5.94	.12	30.03	9.10	6.33	2.75	7.95	.14
	.19	.27	18.59	0.0	18.59	22.30	6.78	.....	2.42	6.85	.28	29.84	9.09	6.75	3.30	9.13	.37
	.95	.14	13.81	0.0	13.81	14.21	3.89	2.26	10.90	9.90	1.20	17.89	4.83	6.34	13.74	12.52	1.51
1	.36	.23	10.51	0.0	10.51	14.15	4.04	1.83	11.34	14.05	3.56	17.52	5.00	5.48	14.28	17.38	4.40
1	.11	.10	12.16	0.0	12.16	14.18	3.97	2.05	11.12	11.98	2.38	17.70	4.92	5.91	14.01	14.95	2.95
2	.33	.05	7.03	0.0	7.03	11.07	3.03	.....	16.46	20.40	6.11	13.79	3.58	5.15	19.02	23.58	7.06
1	.91	.22	6.63	0.0	6.63	10.46	3.22	.....	17.69	20.00	4.21	11.89	3.66	5.28	20.10	22.73	4.77
1	.78	.05	7.79	0.0	7.79	8.51	3.00	.....	16.08	19.90	3.74	10.31	4.34	5.52	19.34	23.95	4.49
1	.80	.04	6.17	0.0	6.17	8.07	3.00	.....	18.95	17.35	2.91	9.45	3.40	5.46	22.20	20.30	3.40
1	.95	.09	6.90	0.0	6.90	9.53	3.19	.....	17.29	19.41	4.24	11.11	3.74	5.35	20.16	22.64	4.93
1	.67	.12	8.66	0.0	8.66	11.07	3.45	.....	15.20	16.96	3.62	13.31	4.14	5.56	18.11	20.23	4.27
	.46	.22	37.81	0.0	37.81	13.70	4.10	2.76	4.41	7.34	1.76	24.33	7.28	7.23	7.83	13.03	3.13
	.45	.21	35.39	0.0	35.39	14.26	4.49	2.01	4.21	6.94	1.78	24.54	7.73	6.61	7.24	11.94	3.06
	.45	.21	36.60	0.0	36.60	13.98	4.29	2.83	4.30	7.14	1.77	24.43	7.50	6.92	7.53	12.48	3.09
	.63	.56	27.19	0.0	27.19	17.75	5.09	0.0	4.87	9.35	1.45	26.85	7.70	6.61	7.36	14.44	2.19
	.73	.33	30.84	0.0	30.84	14.98	4.12	0.0	6.53	10.06	1.82	23.69	6.51	7.64	10.33	15.91	2.87
	.52	.16	27.45	0.0	27.45	16.31	3.96	0.0	4.95	8.42	.92	24.62	6.00	6.61	7.34	12.74	1.39
	.57	.42	28.64	0.0	28.64	16.11	4.72	0.0	5.28	9.60	1.15	24.96	7.31	6.28	8.18	14.88	1.78
	.61	.37	28.53	.....	28.53	16.29	4.47	.....	5.41	9.36	1.33	25.03	6.88	6.78	8.30	14.40	2.05
	.56	.32	31.22	.....	31.22	15.50	4.41	.....	5.04	8.62	1.48	24.83	7.09	6.83	8.05	13.82	2.40

Freed from hulls and with a portion of the fixed oil removed.
These hulls necessarily have a little of the inner seed adhering thereto.
Ground to a fine paste with full content of oil and hulls.

Piesse and Stansell give the following composition of mustard ash:

	White Seeds.		Brown Seeds.
	Yorkshire.	Cambridge.	Cambridge.
Potash	21.29	18.88	21.41
Soda	0.18	0.21	0.35
Lime	13.46	9.34	13.57
Magnesia	8.17	10.49	10.04
Iron oxide	1.18	1.03	1.06
Sulphuric acid	7.06	7.16	5.56
Chlorine	0.11	0.12	0.15
Phosphoric acid	32.74	35.00	37.20
Silica	1.00	1.12	1.41
Sand	1.82	1.95	1.38
Charcoal	12.82	15.14	7.57
	99.85	100.48	99.70

Determination of Myronate of Potassium and Sinapin Sulphocyanate.* —Extract at least 50 grams of the powdered material with several portions of a mixture of equal parts of water and alcohol, digesting with the aid of heat in a flask with a return-flow condenser. Evaporate the alcoholic extract in a tared dish to dryness, and heat at 105° to constant weight. After weighing, incinerate the residue at a temperature sufficiently high to transform to the neutral sulphate the potassium bisulphate resulting from the decomposition of the myronate. The weight of myronate of potassium is obtained by multiplying the weight of neutral sulphate (the final ash) by the factor 4.77. This, deducted from the total weight of the dried alcoholic residue as above, gives that of the sulphocyanate of sinapin.

Determination of Mustard Oil in Mustard Flour.—*Roeser's Method.*† —Mix 5 grams of the sample with 60 cc. of water and 15 cc. of 60% alcohol, and let stand for two hours. Distil into a flask containing 10 cc. of ammonia, and, after about two-thirds of the solution have been distilled off, mix the ammoniacal distillate with 10 cc. of tenth-normal silver nitrate solution, and allow the mixture to stand for twenty-four hours, after which make up with water to 100 cc. Filter, and treat 50 cc. of the filtrate with 5 cc. of tenth-normal potassium cyanide solution. Titrate the excess of cyanide with the tenth-normal silver nitrate, using as an indicator a 5% solution of potassium iodide, made slightly ammoniacal.

* Girard et Duprée Analyse des Matières Alimentaires, p. 678.

† Abs. Analyst, XXVII, 1902, p. 197.

The percentage of mustard oil present is found by multiplying by 2 the number of cubic centimeters of silver nitrate solution taken up by the oil, and multiplying this product by the factor 0.3137.

Microscopical Characteristics of Powdered Mustard.—The principal features of powdered black mustard are represented in Fig. 82. The seed shell or hull is shown in cross-section at (1), *a* being the polygonal-celled epidermis, *b* a layer of palisade-shaped cells, and *c* a thin pigment layer, the brown coloring matter of which is colored blue by iron salts; *d* is the glutinous layer and obscure parenchyma, and *e* the small-celled tissue of the cotyledons, containing fixed oil and albumen.

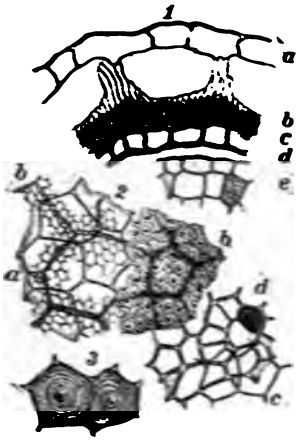


FIG. 82. — Powdered Mustard under the Microscope. ×125. (After Moeller.)

(2) shows in plan view the various layers of the seed shell, the letters of reference corresponding to those of (1).

(3) shows in plan view a bit of the extreme outer mucilaginous layer of the seed-hull.

Fig. 247, Pl XXXII. shows the appearance in water-mount of pure ground mustard. This is a photomicrograph of the ground hulled seed without the extraction of the oil, and should not be taken as a standard for commercial mustard "flour," from which, as a rule, a large portion of the oil has been removed. The cellular tissue of the mustard shown in the form of granular masses of loose, fine gray texture; the

Adulteration of Mustard.—U. S. standards for mustard are as follows: Starch, by diastase method, should not exceed 2.5% and total ash should not exceed 8%.

It is difficult to draw the line between the amount of mustard hulls which may naturally occur in ground mustard, and the excess amount which is sometimes added as an adulterant. Samples in which the patches of hulls predominate in number over the regular cellular tissue of the seed, as seen under the microscope, are undoubtedly adulterated by the fraudulent admixture of ground hulls, that have been separated out from the crushed mustard seeds intended for higher grades. Samples of mustard flour thus adulterated are common.*

In determining starch in mustard, it should be borne in mind that mustard hulls have considerable reducing matter by the diastase process.

The most common adulterants of mustard, other than excess of hulls, are wheat, turmeric, millet and other weed seed, and rice. Yellow, oil-soluble azo-dyes are also employed.

Other adulterants found in Massachusetts have been potato starch, cayenne, corn, and gypsum or "terra alba" (the latter being found in one instance to the extent of 21%).

Fig. 250, Pl. XXXIII, shows a sample of mustard adulterated with wheat bran. Very little besides the adulterant appears in this field.

The common practice of adulterating mustard with wheat is an outgrowth of the old notion that a certain amount of wheat flour was necessary to prevent lumping.

Samples of cheaper mustard flour are occasionally found to contain small amounts of wheat and foreign starch, apparently of accidental occurrence. This is undoubtedly due to the fact that in some localities wild mustard often grows in the wheat-fields, so that after the wheat crop has been harvested, the mustard is gathered and sold. Such mustard seed would naturally contain varying admixtures of wheat, and sometimes seeds of various weeds, which it would not be profitable to separate out, even

* It is claimed by some manufacturers that the hulls thus removed are not used as an adulterant of cheaper mustard flours, in view of the fact that it is difficult or impossible to grind them finely enough, but that they are used up in the manufacture of compound mustard pastes. A sample of ground mustard was recently found by the writer, in which it was noticed that a large number of yellow lumps were distributed through it. These lumps were picked out, transferred to the microscope slide, and crushed and rubbed out under the cover-glass. Examined under the microscope, they were found to consist entirely of a mixture of mustard hulls and turmeric, which would seem to show that hulls were present in this case as an adulterant.

if it were possible to do so. Such contaminated mustard enters into the manufacture of the cheapest grades of flour only.

Fig. 249, Pl. XXXIII, shows the flour of the Dakota brown mustard, which is one of the most common of these wild varieties.

Detection of Coloring Matter.*—Turmeric is best detected by the microscope (see pp. 351 and 352). Oil-soluble coal-tar dyes should be tested for as in the case of cayenne.

NUTMEG AND MACE.

Nature and Composition.—Both nutmeg and mace occur in the fruit of several varieties of trees of the genus *Myristica*, especially of the *Myristica fragrans* or *Myristica moschata*, belonging to the family *Myristicaceæ*. The nutmeg tree is a native of the Malay archipelago, and grows from 20 to 30 feet high, somewhat resembling the orange tree in appearance. It does not produce flowers till its eighth or ninth year, after which it bears fruit constantly for many years. The fruit is a globular, pendant drupe, about 5 cm. in diameter, of a yellowish-green color, the pericarp of which, when ripe, splits in two, showing within it the kernel, completely surrounded by a fleshy, fibrous aril, or covering of a crimson color. This covering, when dried, furnishes the mace of commerce, while the inner kernel, which is a hard, brown seed, is the nutmeg.

The nutmeg seed or kernel, when gathered, is surrounded by a thick tegument, marked with depressions corresponding to the lobes of the aril or mace, and by a second thin, inner envelope, closely adhering to the seed. The whole seed is dried in the sun for about two months, or by the aid of heat, the tegument becoming separated from the kernel, and by breaking with a hammer is readily removed. The kernels

Liming is alleged to be practiced for the purpose of preventing sprouting of the seed.

NUTMEGS are spheroidal, sometimes nearly spherical, from 20 to 25 mm. long and 15 to 18 mm. in diameter. The outer surface is somewhat furrowed. The interior is composed of hard albumen, grayish brown in color, and oily. A cross-section of the kernel presents the appearance of numerous wavy, dark-brown lines on the surface of the section. Near the end of the nutmeg, which is attached to the stem, is a small cavity, in which is the undeveloped embryo, with two cotyledons.

Nutmeg contains a considerable amount of fixed oil, a volatile oil, starch, and albuminous matter. Its volatile oil is colorless, and is soluble in three parts of strong alcohol. The specific gravity of nutmeg oil varies between 0.865 and 0.920, and its specific rotary power $(\alpha)_D = 14$ to 28.

Richardson's analyses of three samples of nutmeg are as follows:

	Water.	Ash.	Volatile Oil.	Fixed Oil or Fat.	Starch, etc.	Crude Fiber.	Albuminoids.	Nitrogen.
Whole limed	6.08	3.27	2.84	34.37	36.98	11.30	5.16	.83
Ground limed	4.19	2.22	3.97	37.30	40.12	6.78	5.42	.87
Ground	6.40	3.15	2.90	30.98	41.77	9.55	5.25	.84

König gives the following minimum and maximum composition of nutmeg:

	Minimum.	Maximum.
Water	4.2	12.2
Albuminoids	5.2	6.1
Volatile oil	2.5	4.0
Fat	31.0	37.3
Carbohydrates	29.9	41.8
Cellulose	6.8	12.0
Ash	2.2	3.3

Winton, Ogden, and Mitchell analyzed four samples of nutmeg of known purity, the following being maximum and minimum results:

	Moisture.	Ash.			Ether Extract.	
		Total.	Soluble in Water.	Insoluble in HCl.	Volatile.	Non-volatile.
Maximum	10.83	3.26	1.46	0.01	6.94	36.94
Minimum	5.79	2.13	0.82	0.00	2.56	28.73

	Alcohol Extract.	Reducing Matters by Acid Conversion, as Starch.	Starch by Diastase.	Crude Fiber.	Nitrogen × 6.25.	Total Nitrogen.
Maximum.....	17.38	25.60	24.20	3.72	7.00	1.12
Minimum.....	10.42	17.19	14.62	2.38	6.56	1.05

Microscopical Structure of Nutmeg. (Fig. 83.)—The thin-walled cells of the parenchyma of the endosperm or albumen are shown at (1), with starch grains. Simple and compound granules of the starch are shown at (2). Aleurone grains appear as shown at (3), and (4) represents in plan view the epidermis, or brown seed coat, with its numerous layers of flat cells. Powdered nutmeg under the microscope in water-mount shows most commonly a sponge-like, loose meshwork of bruised or broken cellular tissue, with many starch granules, and occasional fragments of the epidermis.

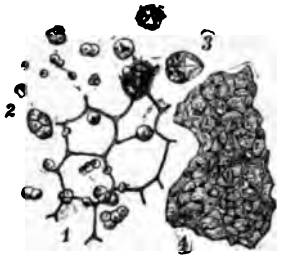


FIG. 83.—Powdered Nutmeg under the Microscope. × 125. (After Moeller.)

Fig. 240, Pl. XXX, is a photomicrograph of a water-mounted sample of pure nutmeg. The starch granules of nutmeg are different from other starches in appearance, being almost circular as a rule, quite uniform in size (averaging 0.006 mm. in diameter), and having very distinct central hyl-

Adulteration of Nutmeg. — The U. S. standards for nutmegs

An inferior variety is known as the Macassar nutmeg. This lacks much of the agreeable pungency of the better grades.

MACE.—The crimson-colored aril that surrounds the nutmeg kernel within the pericarp, as above described (p. 360), has many narrow, flattened lobes. In the process of drying to form the mace of commerce, it loses its brilliant red color, and turns a yellowish brown. When dried, it is brittle, somewhat translucent, and of a pungent odor. Whole mace appear on the market in the form of flat membranous masses, 3 to 4 cm. long.

It contains no starch as such, but has a modified form of starch known as amylodextrin. This is a carbohydrate, $C_{36}H_{62}O_{31}+H_2O$, which produces with iodine a red coloration. Mace has a large amount of fixed oil, as well as considerable resinous and albuminous matter, and a volatile oil which much resembles that of nutmeg.

The specific gravity of volatile oil of mace is rather higher than that of nutmeg oil. Its specific rotary power, $(\alpha)_D = 10$ to 20.

König's figures for the composition of mace are as follows:

	Minimum.	Maximum.
Water	4.9	17.6
Albuminoids	4.6	6.1
Volatile oil	4.0	8.7
Fat	18.6	29.1
Carbohydrates	41.2	44.1
Cellulose	4.5	8.9
Ash	1.6	4.1
Alcoholic extract	45.1	55.7

Richardson gives the following as the results of analyses of three samples made by him:

	Water.	Ash.	Volatile Oil.	Resin.	Undetermined.	Crude Fiber.	Albuminoids.	Nitrogen.
Whole mace	5.67	4.10	4.04	27.50	41.17	8.93	4.55	.73
Ground mace	4.86	2.65	8.66	29.08	35.50	4.48	6.13	.98
Ground mace	10.47	2.20	8.68	23.33	34.68	6.88	5.08	.81

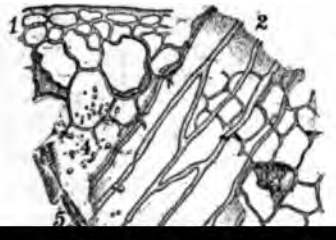
Winton, Ogden, and Mitchell's analyses of fonr samples of pure Banda or Penang mace, as well as of Bombay and Macassar mace, are summarized as follows:

	Moisture.	Ash.			Ether Extract.	
		Total.	Soluble in Water.	Insoluble in HCl	Volatile.	Non-volatile.
True mace: Maximum....	12.04	2.54	1.33	0.21	8.65	23.72
Minimum	9.78	1.81	1.06	0.00	6.27	21.63
Average.....	11.05	2.01	1.13	0.07	7.58	22.48
Macassar.....	4.18	2.01	1.11	0.03	5.89	53.54
Bombay (adulterant)	0.32	1.98	1.37	0.07	4.65	59.81

	Alcohol Extract.	Reducing Matters by Acid Conversion, as Starch.	Starch by Diastase.*	Crude Fiber	Nitrogen X6.25.	Total Nitrogen.
True mace: Maximum....	24.76	34.42	30.43	3.85	7.00	1.12
Minimum	22.07	26.77	23.12	2.94	6.25	1.00
Average.....	23.11	31.73	27.87	3.20	6.47	1.03
Macassar.....	32.89	10.39	8.78	4.57	7.00	1.12
Bombay (adulterant)	44.27	16.20	14.51	3.21	5.06	0.81

* The figures in this column do not express starch, but amylo-dextrin, which like starch may be determined by the diastase method.

Microscopical Structure of Mace.—Fig. 84 shows characteristics of mace, (1) being a cross-section through it, (2) a plan view of the epidermis, showing its elongated, often nearly rectangular cells, and (3) the large-celled parenchyma, in which are numerous oil globules. The contents of the parenchyma cells are for the most part colorless, consisting of albumen, fat, and granules of amylodextrin, which are shown at (4). At (5) are shown fragments of



3% of total ash; nor more than 0.5% ash insoluble in hydrochloric acid; nor more than 10% of crude fiber.

Turmeric and cereal starches are not uncommonly found in mace, but by far the most common adulterant is the so-called false, or wild mace, otherwise known as Bombay mace.

Bombay Mace (*Myristica fatua*) is almost entirely devoid of odor or taste, being nearly as inert as so much starch. It is most properly regarded as an adulterant from its lack of pungency, even though in a sense it is a variety of mace.

Its non-volatile ether extract is twice as high as that of Penang mace, and at room temperature the fixed oil of Bombay mace is a thick and viscous fat, while that of Penang and other maces is a thin oil.

The refractive indices of the fixed oils of various species of pure, as well as of Bombay mace, as determined by Lythgoe in the writer's laboratory, are as follows:

		n_D at 35° C.
Banda Mace	(1)	1.4848
" "	(2)	1.4747
" "	(3)	1.4829
Batavia Mace	(1)	1.4893
" "	(2)	1.4975
Papua Mace	(1)	1.4816
" "	(2)	1.4795
West Indian Mace	(1)	1.4766
Bombay Mace	(1)	1.4615
" "	(2)	1.4633

The microscope indicates at once when Bombay mace is present in a sample. The oil glands situated in the outer layers of Bombay mace are very strongly colored, and contain a deep-red, resinous substance, very different from anything to be found in true mace. The glands of the more interior layers of wild mace have, moreover, a balsam-like substance of a bright-yellow color. In powdered Bombay mace, when mounted in water, nearly every field shows both the red and the yellow lumps in considerable number.

Hefelmann's Test for Bombay Mace * consists in saturating a strip of filter-paper with an alcoholic solution of the mace, and removing the excess of liquid by pressure between filter-paper. On treating with a

* Pharm. Zeit., 1891.

drop of dilute sodium or potassium hydroxide solution, a red color is produced in presence of the wild mace.

Turmeric is tested for chemically as on p. 353.

Macassar Mace is sometimes designated as wild mace, but it is by no means as inert as the Bombay variety, and possesses a slight mace-like odor. Its taste, while distinctive, is not that of true Penang mace. It is distinctly an inferior article, and its volatile oil content, as shown by the analyses on p. 364, is considerably below the minimum for true mace.

REFERENCES ON SPICES.

(See also References on the Microscope, p. 12.)

ARNST, T., and HART, F. Zusammensetzung einiger Gewürze. Zeits. für Angew. Chem., 1893, 136.

BEYTHIEN, A. Einige Paprika-Analysen. Zeits. für Unters. der Nahr. u. Genuss., 5, 1902, 858.

BUSSE, W. Ueber Gewürze. I. Pfeffer. II. Muskatnüsse. III. Macis. Arbeit. a. d. Kais. Gesundheits., 1894, 9, 509; 1895, 11, 390; 1896, 12, 628.

DELAITE, J. Untersuchung von Senfmehl. Rev. Int. des Falsif., 1897, 10, 37.

DYER, U. GILBARD. Unterscheidung zwischen unverfälschtem und extrahirtem Ingwer. Chem. Ztg., 1893, 17, 838.

FORSTER, A. Ueber Gewürze. Zeits. f. öffentl. Chem., 1898, 4, 626.

GÉNIN, V. Epices et Aromates. Analyse des Matières Alimentaires. Girard et Dupré, Paris, 1894.

GICHARD, B. Verfälschung von Zimmetrindenpulver. Zeits. f. Nahr. Hyg. Waarenk., 1895, 9, 281.

HANAUSEK, T. F. Zur Charakteristik des Cayenpfeffers. Zeits. für Nahr. Unters. Hyg., 1893, 7, 297.

—— Verfälshung von Gewürzen. Zeits. für Nahrungsm. Unters. u. Hyg., 1894, 8, 95.

—— Gewürzfälschungen. Apoth. Ztg., 1894, 582.

RICHARDSON, C. Spices and Condiments. U. S. Dept. of Agric., Div. of Chem., Bul. 13, part 2, 1887.

ROETTGER, H. Die Gewürznelken, ihre Verfälschung und Beurtheilung. Ber. XI. Vers. d. freien Verein bayr. Vertreter d. angew. Chem. in Regensburg, 1892, S. 66.

SOLSTEIN, P. Banda und Bombay Maces. Pharm. Ztg., 1893, 454, 467.

SPÄTH, E. Neue Verfälschungen von Gewürzen. Forsch. über Lebensm., 1896, 3, 308.

—— Zur mikroskop. Prüfung des Piments. Forsch. über Lebensm., 1895, 2, 419.

VOGL, A., and HANAUSEK, T. F. Untersuchung der Gewürze. Sudd. Apoth. Ztg., 1896.

WAAGE, T. Banda und Bombay Maces. Pharm. Centralbl., 1892, 33, 372.

WARBURG, O. Die Muskatnuss, ihre Geschichte, Botanik, Kultur, u. s. w. Leipzig, 1897.

WEIGLE, T. Untersuchungen über die Zusammensetzung des Pfeffers. Ber. Pharm. Ges., 1893, 210.

WINTON, A. L. Spices. U. S. Dept. of Agric., Bur. of Chem., Bul. 65, 1902.

Conn. Exp. Sta. An. Rep. 1898, 1899.

Mass. State Board of Health An. Reports, 1883 et seq.

CHAPTER XII.

EDIBLE OILS AND FATS.

Nature and Properties.—The oils and fats are the glycerin salts or glycerides of the fatty acids, the most important, on account of their occurrence in nearly all fats and oils, being the triglycerides of oleic, palmitic, and stearic acids, known as olein, palmitin, and stearin, respectively.

Fats and oils are insoluble in water, and are almost insoluble in cold 95% alcohol, though they are somewhat soluble in absolute alcohol. They are readily soluble in ether, petroleum ether, chloroform, amyl alcohol, oil of turpentine, and carbon bisulphide.

Following is a list of the fatty acids whose glycerides are found in edible oils and fats, together with their melting- and boiling-points when these have been determined, and the oils and fats in which they occur.

ACIDS OF THE ACETIC SERIES $C_nH_{2n}O_2$.*

Name.	Formula.	Melting-point	Boiling-Point	Occurrence in Oils and Fats.

ACIDS OF THE OLEIC SERIES $C_nH_{2n-2}O_2$.

Name.	Formula.	Melting-point.	Boiling-point.	Occurrence in Oils and Fats.
Hypogæic.....	$C_{16}H_{30}O_2$	33°	236°	Peanut.
Oleic.....	$C_{18}H_{34}O_2$	14°	232.5°	Nearly all fats and oils.
Iso-oleic*....	$C_{18}H_{34}O_2$	44-45°	....	
Rapic.....	$C_{18}H_{34}O_2$	....	....	Rape and mustard.
Erucic.....	$C_{22}H_{42}O_2$	33-34°	264°	" " "

ACIDS OF THE LINOLEIC SERIES $C_nH_{2n-4}O_2$.

Name.	Formula.	Melting-point.	Boiling-point.	Occurrence in Oils and Fats.
Linoleic.....	$C_{18}H_{32}O_2$	Under −18°C.	.....	Olive, cottonseed, peanut, sesame, cocoa butter, poppy seed, sunflower.

* Solid oleic acid.

Saponification of Fats and Oils.—By this term is meant the decomposition of the glycerides composing the fats or oils, whereby the triatomic alcohol glycerin and the fatty acids are separated. The saponification process is commonly applied in carrying out many determinations of value on fats and oils, such as those of the soluble and insoluble fatty acids, the Reichert value, etc. As commonly carried out, the triglycerides are first split up into glycerin and the soluble soaps of the fatty acids by the action of caustic alkali, usually in solution in alcohol. This part of the process in the case of a given oil, composed, for example, of stearin, olein, and palmitin, is illustrated as follows:

(1) $\underset{\substack{\text{Stearin or}\\\text{triglyceryl}\\\text{stearate}}}{C_3H_5(C_{18}H_{35}O_2)_3} + 3KOH = \underset{\text{Glycerin}}{C_3H_5(OH)_3} + 3K(\underset{\substack{\text{Potassium}\\\text{stearate}}}{C_{18}H_{35}O_2})$

(2) $\underset{\substack{\text{Palmitin or}\\\text{triglyceryl}\\\text{palmitate}}}{C_3H_5(C_{16}H_{31}O_2)_3} + 3KOH = C_3H_5(OH)_3 + 3K(\underset{\substack{\text{Potassium}\\\text{palmitate}}}{C_{16}H_{31}O_2})$

(3) $\underset{\substack{\text{Olein or tri-}\\\text{glyceryl oleate}}}{C_3H_5(C_{18}H_{33}O_2)} + 3KOH = C_3H_5(OH)_3 + 3K(\underset{\substack{\text{Potassium}\\\text{oleate}}}{C_{18}H_{33}O_2})$

These "soaps," or potassium salts of the fatty acids, are further decomposed by the action of sulphuric acid into the free fatty acids and potassium sulphate, in the case of potassium stearate, as follows:

$$\underset{\text{Potassium stearate}}{2K(C_{18}H_{35}O_2)} + H_2SO_4 = K_2SO_4 + 2H(\underset{\text{Stearic acid}}{C_{18}H_{35}O_2})$$

Analysis of Edible Oils and Fats.—No class of food products presents more difficulties in the analyst than the fats and oils, in that the various physical and chemical constants by which one derives information as to their nature or purity differ within such wide limits that it is not easy to prescribe absolute standards. Many elements enter in to cause this variation, chief among which are, in vegetable oils, the large number of varieties of fruits or seeds from which each oil is in different localities obtained, as well as the various grades of each oil with respect to refining. In the animal fats, butter and lard, the quality of the food on which the animal is reared undoubtedly influences the chemical constants of the fat, and in all fats and oils much depends upon their age, and the conditions under which they are kept as to temperature, exposure to light and air, etc.

Rancidity is undoubtedly an oxidation process, due to the action of air and light rather than to that of ferments or micro-organisms, as was formerly supposed. It seriously affects various constants, and care should be taken by the analyst to keep all samples in a dark, cool place and as far as possible in tight containers.

As a rule rancidity develops more readily in the liquid oils in which the olein predominates than in the solid fats, which are composed more largely of palmitin and stearin.

Judgment as to Purity of a given oil or fat should not be hastily given. It is sometimes comparatively easy to prove the presence and approximate amount of an adulterant, the various constants all serving to identify it without fail. Again, in some cases it is easy to pronounce the sample adulterated, without being able to definitely state the exact nature of the adulterant. The tests to be employed depend on the particular case in hand. Sometimes the determination of two or three constants

A steam- or hot-water-jacketed funnel as represented in Fig. 85 is convenient for filtering fats, or, in the absence of this contrivance for keeping the fat in a molten condition, a hot funnel may be employed, the filtering being best conducted in a warm closet or oven.

Portions of the fat for the various determinations may be measured off with a pipette while the fat is still hot, but a much better way is to

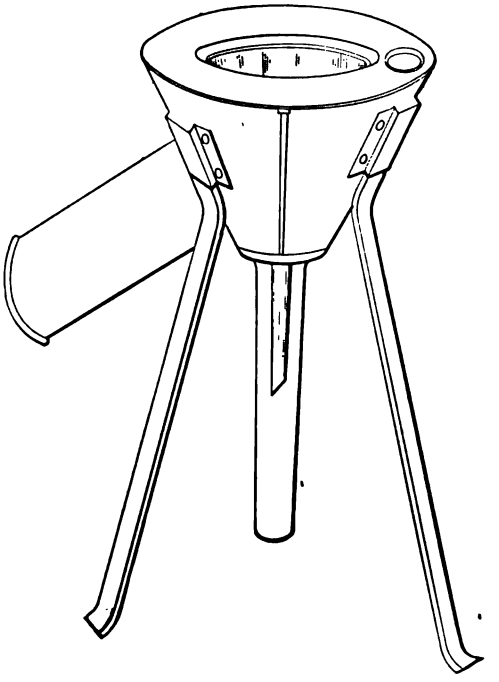


FIG. 85.—Jacketed Funnel for Hot Filtration.

cool the fat (over ice if necessary), and to weigh the desired amounts in the solid state. This can very readily be done by placing a flat platinum or other dish on the scale-pan, covering it with a moderately thick, cut filter-paper somewhat larger in diameter than the dish and designed to lie flat upon it, and taking the tare of both dish and filter. The solidified fat, after mixing with a stirring-rod, is transferred in one or more portions to the middle of the filter, and the exact weighed amount is obtained, after which, by carefully handling the edges of the filter and folding in the latter, the fat with the filter may be transferred to a flask or other receptacle.

SPECIFIC GRAVITY.—The specific gravity of liquid oils is most conveniently taken either at room temperature or at 15.5°, being always

best referred to the latter. Either the hydrometer, Westphal balance, Sprengel tube, or pycnometer are employed, according to the degree of accuracy required. If taken at any other temperature than 15.5°, say at room temperature, T, the specific gravity may be computed at 15.5° by the formula

$$G = G' + K(T - 15.5),*$$

in which G is the specific gravity at 15.5°, G' the specific gravity at $T°$, and K a factor varying with the different oils as follows:

FACTORS FOR CALCULATING SPECIFIC GRAVITY.

Oil.	Correction for 1° C.	Observer.
Cod-liver oil.	0.000646	A. H. Allen
Lard oil.	.000658	C. M. Wetherill
Olive oil.	.000629	C. M. Stillwell
Peanut oil.	.000655	A. H. Allen
Rape oil.	.000620	"
Sesame oil.	.000624	"
Cottonseed oil.	.000629	"

Unless the most accurate work is necessary, it is sufficient to assume in all cases $K = 0.00064$, in which case the formula becomes $G = G' + 0.00064(T - 15.5)$.

In the case of solid fats, it is most convenient to take the specific gravity of the melted fat. This may be done at any temperature above the melting-point by either of the instruments above described, or at the temperature of boiling water by the Westphal balance or pycnometer.

When the pycnometer is used, it is immersed in a water-bath, the temperature of which is well above the melting-point of the fat, say 25°

FACTORS FOR CALCULATING SPECIFIC GRAVITY.

Fats.	Correction for 1° C.
Cocoa butter	0.000717
Tallow	.000675
Lard	.000650
Butter fat	.000617
Cocoanut stearin	.000674
Cocoanut oil	.000642
Palm nut oil	.000657

Either the Westphal balance or the hydrometer may be used directly on the melted fat, carefully recording the temperature and calculating as above.

For making the determination with the Westphal balance at the temperature of boiling water, the melted fat is contained in a vessel immersed in a boiling water-bath, and kept sufficiently long to acquire that temperature, which is carefully noted.

*A. O. A. C. Method.**—The pycnometer, being perfectly clean, is first weighed with the stopper, after which it is filled with freshly boiled, hot, distilled water and placed in a bath of boiling water, where it is kept for half an hour, replacing any loss by evaporation in the flask with boiling distilled water. The stopper of the pycnometer, previously heated at 100°, is then inserted, and the flask removed and wiped perfectly dry. It is then allowed to cool nearly to room temperature, and weighed on the balance when the temperature is the same as that of the room.

The flask, being again perfectly clean and dry, is filled while hot with freshly melted and filtered hot fat, free from air-bubbles, and kept for half an hour in a boiling water-bath, after which the stopper, previously heated as before to 100°, is inserted, and the flask taken from the bath and wiped dry. It is then allowed to cool and weighed when the temperature of the room has been reached.

The specific gravity is calculated by dividing the weight of the fat by the weight of the water previously found.

Having once obtained the weight of the flask and the weight of a volume of water contained therein when at boiling temperature, these figures can be constantly used without redetermination, if the flask is cleaned thoroughly each time.

Calculation of Proportions of Two Known Oils in Mixture.†—This may be roughly accomplished from the specific gravity of the mixture and of the oils known to compose it.

* U. S. Dept. of Agric., Bur. of Chem., Bul. 65, p. 21.

† Villiers et Collin, Les Substances Alimentaires, p. 646.

Let G = specific gravity of mixture,
D and D' = specific gravity of the two oils,
and X = % oil of specific gravity D.

Then $X = \frac{100(G-D')}{D-D'}$.

EDIBLE OILS AND FATS ARRANGED IN ORDER OF SPECIFIC GRAVITY.

	Specific Gravity.
Cocoa butter	.976 to .950
Mutton tallow	.953 " .937
Beef "	.952 " .943
Butter	.940 " .926
Lard	.938 " .934
Poppyseed oil	.927 " .924
Sunflower oil	.926 " .924
Corn oil	.926 " .921
Cottonseed oil	.925 " .922
Sesame oil	.924 " .923
Peanut oil	.921 " .917
Mustard oil	.920 " .916
Olive oil	.918 " .916
Rape oil	.917 " .913

The Viscosity, or degree of fluidity in the case of edible oils, is of less importance than in the case of lubricating oils, and gives little insight into the nature or purity of the sample.

Hence a discussion of various viscosimeters and their use will not be included here, but reference is made to Lewkowitsch * for information regarding them.

Melting-point.—A piece of small glass tubing is drawn out to a capillary open at both ends, and this is inserted into a beaker of the fat, melted at a temperature slightly above its fusing-point. A portion of the melted

level of the water in the flask, as shown. The water in the flask is then heated *very gradually*, so that the rise of temperature as shown by the thermometer does not exceed 0.5° C. per minute, the exact temperature at which fusion of the fat occurs being recorded as the melting-point.

The flame is then removed, and the temperature at which the fat solidifies is noted as the solidifying-point.

The mean of two or three determinations is usually taken as the true melting and solidifying-points.

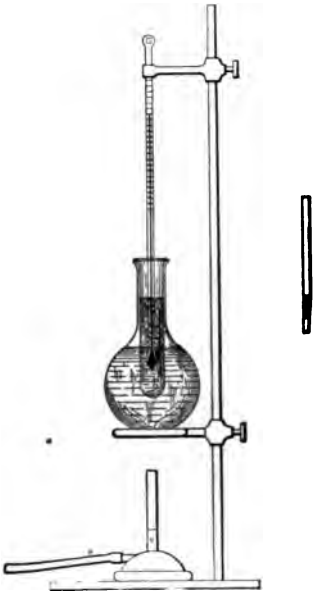


FIG. 86.

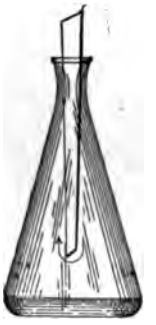


FIG. 87.

FIG. 86.—Apparatus for Determining Melting-point. Capillary tube with enclosed fat shown on the right, enlarged.

FIG. 87.—Reichert Flask with Card Inserted for Quick Evaporation.

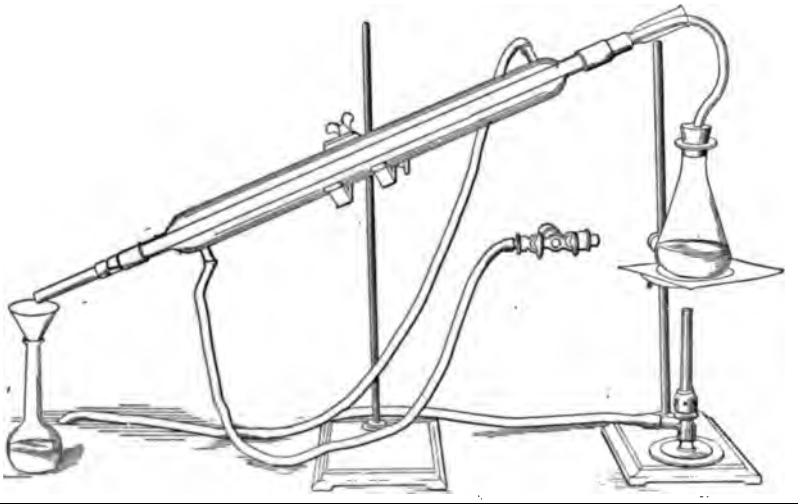
REICHERT-MEISSL PROCESS FOR VOLATILE FATTY ACIDS.—This process has undergone various modifications from time to time. Reichert originally used 2.5 grams of fat, but Meissl, who improved the process, used 5 grams, so that the Reichert-Meissl number is now expressed on the basis of 5 grams of fat. The method is conveniently carried out as follows:

Five grams of the fat are transferred to a dry, clean Erlenmeyer flask of about 300 cc. capacity, 10 cc. of 95% alcohol are added, and 2 cc. of sodium hydroxide solution (prepared by dissolving 100 grams of sodium hydroxide in 100 cc. of water). The flask with its contents is then heated

on a water-bath with a funnel in the neck, which satisfactorily replaces the return-flow condenser originally prescribed. The heating is continued with occasional shaking till saponification is complete. This stage of the process is indicated by the appearance of the solution, which is then perfectly clear and free from fat globules.

The condenser-funnel being removed, the contents of the flask are next evaporated by continued heating over the bath to dryness. This may be hastened by inserting a card in the neck of the flask, as shown in Fig. 87, thus starting a circulatory movement to the air through the flask.

The dry soap thus formed is then dissolved by warming on the water-bath with 135 cc. of added water, shaking the flask occasionally. After



tion is conducted by so grading the heat that the receiving flask is filled with the distillate in about thirty minutes.

Finally the entire distillate is titrated with decinormal sodium hydroxide, using 0.5 cc. of a solution of phenolphthalein as an indicator. The number of cubic centimeters of decinormal alkali required to neutralize the acidity of the distillate from 5 grams of the fat in the manner described expresses what is known as the Reichert-Meissl number.

*Leffmann and Beam's Modification.**—Five grams of the fat placed in the flask are treated with 20 cc. of a solution of soda in glycerin (20 cc. of a 50% solution of sodium hydroxide in 180 cc. of glycerin), heating the flask till the contents are completely saponified. The solution becomes perfectly clear, showing complete saponification in about five minutes, after which 135 cc. of water are added to the clear soap solution, at first drop by drop to prevent foaming; 5 cc. of the dilute sulphuric acid are then added, and the distillation conducted at once without first melting the fatty acids.

EDIBLE OILS AND FATS IN THE ORDER OF THEIR REICHERT-MEISSL. NUMBER.

	Lowest.	Highest.	Average.
Butter	24.5	32	28.25
Cocoanut oil	6.65	7.8	7.2
Cocoa butter	....	....	3.2
Corn oil	1.32	5.0	3.16
Lard	....	....	1.10
Cottonseed oil	....	....	0.95
Sesame oil	0.70	1.20	0.95
Rape oil	0.58	0.90	0.74
Olive oil	....	....	0.60
Beef tallow	....	....	0.5

Determination of Soluble and Insoluble Fatty Acids.—*A. O. A. C. Method.*†—*Soluble Acids.*—Five grams of the fat are weighed out and transferred to an Erlenmeyer flask of the same size and in the same manner as that used for the Reichert-Meissl process. 50 cc. of alcoholic potash solution are added (40 grams of potassium hydroxide in 1 liter of 95% redistilled alcohol) and the flask, provided with a return-flow condenser, is heated on the water-bath till saponification is complete, as evidenced by the clear solution free from fat globules. The alcoholic solution of potash is preferably measured from a pipette, from which it is allowed to drain for a noted interval of time, say thirty seconds.

* Leffmann and Beam, Select Methods of Food Analysis, p. 146.

† U. S. Dept. of Agric., Div. of Chem., Bul. 46, p. 47.

After complete saponification, the condenser is removed and the alcohol is evaporated by further heating. One or more blanks are prepared at the same time, using the same 50-cc. pipette for measuring, and applying the same time limit for draining the pipette. The blanks are first titrated, after evaporation, with half-normal hydrochloric acid, using phenolphthalein as an indicator. Then add to the flask containing the fatty acids 1 cc. more of the half-normal acid than is found necessary to neutralize the alkali in the blanks, after which the flask is again heated with a funnel in the neck till the fatty acids have completely separated in a layer on top of the solution. Then cool the flask in ice water till the fatty acids are solidified, after which decant the liquid portion through a filter, previously dried in the oven and weighed, into a liter flask, keeping the solid mass of fatty acids intact. Next add 200 or 300 cc. of hot water to the flask containing the fatty acids, and again melt over the water-bath till they collect as before on top, having again inserted the funnel to act as a condenser, and occasionally shaking the contents of the flask during heating. Cool as before in ice water, after which again decant the liquid from the solid mass through the same filter into the liter flask. Repeat this process of washing, melting, cooling, and decanting three times, receiving all the wash water through the same filter in the same flask. Make up the washings with water to the liter mark, and, after mixing, two portions of 100 cc. each are titrated with tenth-normal sodium hydroxide, using phenolphthalein for an indicator. Each reading is multiplied by ten to represent the total volume, and the figure thus obtained represents the number of cubic centimeters of tenth-normal alkali necessary to neutralize the acidity of the soluble fatty acids, together with the excess of half-normal acid used, amounting to 1 cc.



cator, and weighed. After once heating for two hours, cooling, and weighing, heat again for half an hour, cool, and weigh. If a considerable loss in weight is found, heat for an additional half-hour. It is best, however, to avoid too prolonged heating, lest oxidation of the fatty acids should produce an increase in weight.

Hehner's Method.—Transfer the fatty acids left in the original Erlenmeyer flask to the thoroughly wet, tared filter, washing out the flask with hot water, thus bringing all the fatty acids upon the filter, which, if of good quality and thoroughly wet beforehand, will retain them. If, however, oily particles are noticed in the filtrate, they may be solidified by cooling in ice water, and afterwards removed by a glass rod and transferred to the filter. After draining dry, the funnel is immersed in cold water to solidify the fatty acids, and the filter containing them is transferred to a weighed dish, which is dried for two hours in the oven at 100°, cooled in the desiccator, and weighed, subtracting the weight of the dish and filter.

EDIBLE OILS AND FATS ARRANGED IN ORDER OF INSOLUBLE FATTY ACIDS.

Mustard oil	96.2 to 95.1
Cottonseed oil	96 " 95
Corn oil	96 " 93
Lard	96 " 93
Peanut oil	95.8
Sesame oil	95.7
Beef tallow	95.6
Mutton tallow	95.5
Poppyseed oil	95.2 " 94.9
Rape oil	95.1
Sunflower oil	95
Olive oil	95
Cocoa butter	94.6
Cocoanut oil	90 " 88.6
Butter	89.8 " 86.5

Saponification Number. — *Koettstorfer's Method.* — By the saponification number is meant the number of milligrams of potassium hydroxide necessary to completely saponify 1 gram of the fat. Between 1 and 2 grams of the fat are transferred in the usual manner (see p. 371) to an Erlenmeyer flask, and 25 cc. of the alcoholic potash solution (40 grams of potassium hydroxide in 1 liter of 95% alcohol) are added with a graduated pipette, which is allowed to drain for a noted period of time, say thirty seconds. The determination should preferably be made in duplicate.

Conduct the saponification as in the case of the soluble fatty acids by heating on the water-bath. After saponification, remove from the bath, cool, and titrate with half-normal hydrochloric acid, using phenolphthalein as an indicator. Titrate also several blanks in which 25 cc. of the alcoholic potash solution are measured out with the same pipette as before, and allow to drain for the same amount of time. Subtract the number of cubic centimeters of half-normal acid necessary to neutralize the alkali in the case of the saponified fat from that necessary to neutralize the blank, multiply the result by 28.06, and divide the product by the number of grams of fat taken.

EDIBLE OILS AND FATS ARRANGED IN ORDER OF THEIR SAPONIFICATION NUMBER.

	Minimum.	Maximum.	Mean.
Cocoanut oil	240.2	268.4	257.3
Butter	225	230	227.5
Cocoa butter	192	202	197
Beef tallow	193.2	200	196.6
Lard	195.3	196.6	196
Lard oil	195	196	195.5
Cottonseed stearin	194.6	195.1	194.8
Poppyseed oil	190	198	194
Cottonseed oil	191	196.6	193.8
Peanut oil	190	197	193.5
Sunflower oil	193	194	193.5
Sesame oil	187.9	193.4	192.2
Olive oil	185	196	191.5
Corn oil	188	193.4	190.7
Rape oil	170.2	179.2	174.2
Black mustard oil	174	174.9	174.3
White mustard oil	170.3	173.4	172.8

THE IODINE ABSORPTION NUMBER.—This determination is based on

(3) *Starch paste*, prepared by boiling 1 gram of starch in 200 cc. of water for ten minutes, then cooling.

(4) *Potassium Iodide Solution*, made by dissolving 150 grams of the salt in water, and making up the volume to 1 liter.

(5) *Potassium Bichromate Solution* for standardizing the thiosulphate, made by dissolving 3.874 grams of chemically pure potassium bichromate in distilled water, and making up the volume to 1 liter.

The sodium thiosulphate solution is standardized as follows: 20 cc. of the potassium bichromate solution are introduced into a glass-stoppered flask together with 10 cc. of potassium iodide and 5 cc. of strong hydrochloric acid. Then slowly add from a burette the sodium thiosulphate solution, till the yellow color of the solution has nearly disappeared, after which a little of the starch paste is added, and the titration carefully continued to just the point of disappearance of the blue color. The reaction which takes place is as follows:

$$K_2Cr_2O_7 + 14HCl + 6KI = 2CrCl_3 + 8KCl + 6I + 7H_2O.$$

The equivalent of 1 gram of iodine in terms of the thiosulphate solution is found by multiplying the number of cubic centimeters of the latter solution required for the above titration by 5.

If, for example, 16.4 cc. of the thiosulphate solution are required for 20 cc. of the bichromate solution, then 1 gram of iodine is equivalent to $16.4 \times 5 = 82.0$ cc. of sodium thiosulphate solution, or 1 cc. of the thiosulphate solution $= \frac{1}{82} = 0.0122$ gram of iodine. 1 cc. of exactly decinormal thiosulphate is theoretically equivalent to 0.0127 gram of iodine.

Method of Procedure.—Place 1 gram of the solid fat, or from 0.2 to 0.4 gram of oil, in a glass-stoppered flask or bottle of 300 cc. capacity. In the case of oils, this may conveniently be done by difference, weighing first a small quantity of the oil in a beaker with a short piece of glass tubing to serve as a pipette, transferring a number of drops of the oil from the beaker to the bottle, and again weighing the beaker and contents. The number of drops of oil required for the desired weight is first ascertained experimentally. Dissolve the oil in 10 cc. of chloroform, and after solution has taken place, add 30 cc. of the iodine solution, shake, and set the flask in a dark place for three hours, shaking occasionally. When ready for the titration, add 20 cc. of the potassium iodide solution (the purpose of which is to keep in solution the mercuric iodide formed, which would otherwise precipitate on dilution) and 100 cc. of

distilled water. Titrate the excess of iodine by the thiosulphate solution, which is slowly added from a burette till the yellow color has nearly disappeared, after which, by the addition of a little starch paste, the final few drops of thiosulphate are added to dispel the blue color. Near the end of the reaction the flask should be stoppered and vigorously shaken, in order that all the iodine may be taken up, and sufficient thiosulphate should be added to prevent a reappearance of any blue color in five minutes.

Two blanks are conducted at the same time and in similar flasks or bottles, in exactly the same manner as in the case of the above titration, except that the fat is omitted. This is to get the true value of the iodine solution in terms of the thiosulphate solution.

Suppose, for example, in the case of the blanks, 30 cc. of the iodine solution required in one instance 46.1 cc. of sodium thiosulphate solution, and in the other 46.5 cc. The mean is 46.3. Suppose 15.1 cc. of thiosulphate solution were required for the excess of iodine remaining over and above that absorbed by 1 gram of the fat in the above process. Then the thiosulphate equivalent to the iodine absorbed by the fat would be $46.3 - 15.1 = 31.2$ cc., and the per cent of iodine absorbed would be $31.2 \times 0.0122 \times 100 = 38.06$.

EDIBLE OILS AND FATS ARRANGED IN ORDER OF THEIR HÜBL NUMBER.

	Lowest.	Highest.	Average.
Poppyseed oil	132.6	143.3	138
Sunflower oil	118	133.3	125.7
Corn oil	111.2	123	117.1
Cottonseed oil	108	110	109.5
Sesame oil	103	110	107.5
Rape oil	99	106	102.5

and the length of time required to insure saturation of the oil with the iodine.

Of late two other methods have come into prominence, viz., the Wijs and the Hanus. The reagents in both these methods hold their strength for months without change, and the time required for carrying out the reaction in the case of most of the edible oils and fats is very short.

Of the three methods, that of Hanus has the advantage of greatest simplicity in the composition and preparation of the chief reagent.

Tolman and Munson* have shown that with oils and fats having iodine numbers below 100, the three methods give practically identical figures, while with oils having high iodine numbers, the Wijs and Hanus methods give higher results than the Hübl, but are doubtless more nearly correct.

The following are comparative results of the three methods:*

Number of Analyses.			Hübl's Number (3 hours).	Wijs's Number (30 min.).	Hanus' Number (30 min.).	Difference between Wijs and Hübl.	Difference between Hanus and Hübl.
1	Cocoanut oil.....		8.93	9.05	8.60	+0.12	−0.33
2	Butter—	minimum	34.8	35.9	35.4	+1.1	+0.6
		maximum.	35.3	36.2	35.3	+0.9	+0.0
1	Oleo oil.		42.6	43.5	43.3	+0.9	+0.7
4	Oleomargarine—	minimum	52.5	52.9	52.0	+0.4	−0.5
		maximum.	66.3	66.0	64.8	−0.3	−1.5
2	Lard oil—	minimum	69.3	70.5	69.8	+1.2	+0.5
		maximum.	73.7	74.5	73.9	+0.7	+0.2
36	Olive oil—	minimum	79.2	79.9	80.6	+0.7	+1.4
		maximum.	89.8	91.4	90.0	+1.6	+0.2
		average	84.0	85.3	84.6	+1.3	+0.6
3	Peanut oil—	minimum	94.5	95.2	94.1	+0.7	−0.1
		maximum.	107.7	109.5	107.7	+1.8	+0.0
5	Mustard oil—	minimum	98.4	104.3	103.8	+5.9	+5.4
		maximum.	113.0	118.2	116.8	+5.2	+3.8
2	Rape oil—	minimum	100.2	104.1	102.8	+3.9	+2.6
		maximum.	101.3	105.7	105.2	+4.4	+3.8
1	Sunflower oil		106.4	109.2	107.2	+2.8	+0.8
3	Cottonseed oil—	minimum	103.8	105.3	105.2	+1.5	+1.4
		maximum.	106.2	107.3	107.8	+1.1	+1.6
1	Sesame oil.		106.4	107.0	106.5	+0.6	+0.1
3	Corn oil—	minimum	119.0	122.2	119.6	+3.0	+0.4
		maximum.	123.3	129.2	126.0	+5.8	+2.7
2	Poppyseed oil—	minimum	133.4	135.2	132.9	+1.8	−0.5
		maximum.	134.9	139.1	138.4	+4.2	+3.5

Hanus' Method.†—*Reagents.*—*Iodine Solution.*—Dissolve 13.2 grams of pure iodine in 1 liter of pure glacial acetic acid (99%), and to the cold

* Jour. Am. Chem. Soc., 25 (1903), p. 244.

† Zeits. f. Unters. Nahr. u. Genus., 4 (1901), p. 913. Also Hunt, Jour. Soc. Chem. Ind. 21 (1902), p. 454.

solution add 3 cc. of bromine, or sufficient to practically double the halogen content when titrated against the thiosulphate solution, but with the iodine slightly in excess.

Decinormal Thiosulphate Solution, Starch Paste, and Potassium Iodide Solution, as in Hübl's method.

Method of Procedure.—Proceed as in Hübl's method, substituting 30 cc. of the Hanus iodine reagent for that of Hübl, stirring the solution before adding the water, and, instead of adding 20 cc. of the potassium iodide solution, use only 10 cc.

Only half an hour is required for full saturation of the oil by the iodine in the Hanus method, as against three hours in the Hübl. In case of the non-drying oils and fats, the reaction takes place in from eight to fifteen minutes, though it is best to let the flask set for half an hour at least, in all cases. With oils having an iodine number in excess of 100, Tolman and Munson recommend one hour's standing.

On account of the high coefficient of expansion of acetic acid, care should be taken that the temperature is the same when the iodine solution is measured for the blank and for the determination, as otherwise a serious error may be introduced.

Wijs's Method.*—*Reagents.—Iodine Solution.*—Dissolve 13.2 grams of pure iodine in 1 liter of pure glacial acetic acid, and pass through the larger portion of this solution a current of carefully washed and dried chlorine gas † until the solution is practically decolorized. Finally add enough of the original solution of iodine in acetic acid to restore the iodine color, so that there is a slight excess of iodine.

Hunt's Modified Iodine Solution.—Dissolve 10 grams of iodine trichloride in 1 liter of pure glacial acetic acid, and finally add and dissolve 10.8 grams of pure iodine.



cottonseed, rape, sesame, corn, and mustard, thirty minutes; for drying oils, such as sunflower and poppyseed, one hour.

The Bromine Index or Bromine Absorption Number.—The measure of the amount of bromine absorbed by the oils and fats is a useful factor. By the bromine index is understood the weight of bromine which is absorbed by 1 gram of a given oil. The bromine index of various oils has been determined as follows:*

	Bromine Index.	Observer.
Poppyseed	0.835	Levallois
Mustard	0.763	Girard
Sesame	0.695	Levallois
Cottonseed	0.645	"
Rape	0.632	Girard
Peanut	0.530	Levallois
Olive	0.500 to 0.544	"

Method of Levallois.—Five grams of the oil are saponified with alcoholic potash in a 50-cc. graduated flask by the aid of a gentle heat. At the end of the saponification and after cooling, the flask is filled to the mark with alcohol, and, after shaking, 5 cc. are removed by means of a pipette and transferred to a flask. A slight excess of hydrochloric acid is added to set free the fatty acids, and from a burette a standardized solution of bromine water is run in till with constant shaking a permanent yellow color persists. The bromine is previously standardized with potassium iodide and sodium thiosulphate. The weight of bromine fixed by 1 gram of the fat is then calculated.

Mill's Method.—Modified.†—Dissolve 0.1 gram of the filtered and dried fat in 50 cc. of carbon tetrachloride or chloroform in a 100-cc. stoppered bottle. From a burette a standard solution of bromine in carbon tetrachloride, approximately tenth-normal (8 grams to a liter), is slowly added to the oil solution till, after fifteen minutes, a permanent coloration remains. The amount of bromine absorbed is calculated by comparing with the color similarly produced in a blank experiment, or an excess of bromine solution may be run in and the solution titrated back with a standard solution of thiosulphate, using potassium iodide and starch.

THERMAL TESTS.—The rise in temperature produced by the action of certain reagents on various oils and fats, when applied in a definite manner, has been found to be of considerable value, especially in the case of sulphuric acid and of bromine.

* Villiers et Collin, Les Substances Alimentaires, p. 680.

† Allen, Commercial Org. Analysis, II, part 1, p. 63.

The Maumené Test,* or thermal reaction with sulphuric acid, is most readily carried out in a beaker of say 150 cc. capacity, which is set into a larger beaker or vessel of any kind, the space between the two being packed with felt or cotton waste. The inner beaker is removed, and into it is weighed 50 grams of the oil. It is then replaced and the packing adjusted, if necessary, after which the temperature of the oil is noted with a thermometer. From a burette containing the strongest sulphuric acid of the same temperature as the oil, 10 cc. are run into the beaker, at the same time stirring the mixture of acid and oil with the thermometer. The temperature rises somewhat rapidly, and remains for an appreciable time at its maximum point, which should be noted. The difference in degrees centigrade between the initial temperature of the oil and the maximum temperature of the mixture expresses the Maumené number.

With certain oils, as cottonseed, considerable frothing ensues when concentrated acid is employed, making an accurate determination of the Maumené number somewhat difficult. In this case it is better to employ a somewhat weaker acid, and to express results in terms of what is called the "specific temperature reaction." This is the result obtained by dividing the rise of temperature in the case of the oil by the rise of temperature in the case of water, using the same strength of acid, and multiplying the quotient by 100. Indeed, it is of importance in all cases to compare results on oils with those obtained by carrying out the same test on water.

Bromination Test.—This test depends upon the avidity with which the oils and fats absorb bromine, the rise in temperature caused by the reaction being measured in this case rather than the actual *amount* of bromine absorbed, as in the case of the iodine absorption. Indeed, there

apparatus employed and in the manner of diluting the oil and applying the reagent. The calorimeter employed by Gill and Hatch,* Fig. 89, is conveniently made and is very satisfactory. It consists of a long, narrow, flat-bottomed tube, held by a cork in a small beaker, in such a manner that it is surrounded by an air jacket. The small beaker is set into one of larger size, the space between the two being packed with cotton waste. Five grams of the oil or fat are dissolved in 25 cc.

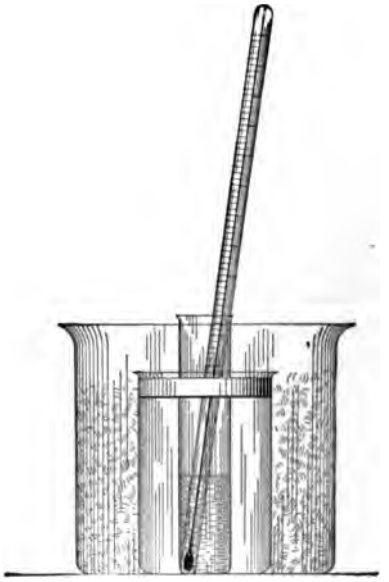


FIG. 89.

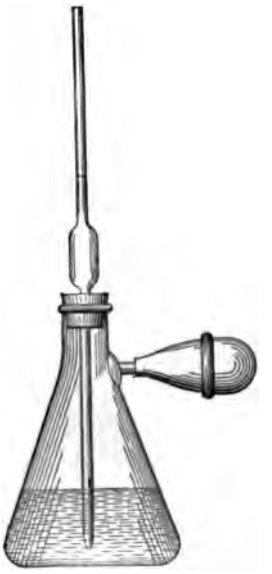


FIG. 90.

FIG. 89.—Gill and Hatch's Calorimeter for the Bromination Test with Oils.
FIG. 90.—Wiley's Pipette for Measuring Bromine in Chloroform.

of chloroform or carbon tetrachloride, and 5 cc. of this solution (containing 1 gram of the oil) are transferred by a pipette to the inner tube of the above calorimeter, being careful not to let it flow down the sides of the tube. The temperature of the oil is then taken by a thermometer graduated to 0.2°. The bromine reagent, which should be freshly prepared, is made up by measuring from a burette one part by volume of bromine into four parts of chloroform or carbon tetrachloride. The reagent is transferred to a measuring-flask devised by Wiley,† consisting of a side-necked filter-flask provided with a per-

* Jour. Am. Chem. Soc., XXI (1899), p. 27. Gill, Oil Analysis, p. 50.
† Jour. Am. Chem. Soc., XVIII (1896), p. 378.

forated rubber stopper into which the stem of a 5-cc. pipette is fitted, Fig. 90. A bulb on the side-neck serves to fill the pipette. This pipette, filled to the mark with the bromine reagent (which should be at the same temperature as the oil solution in the calorimeter), is first covered by the finger and removed, and its contents of 5 cc. allowed to flow down the sides of the inner tube of the calorimeter and mingle with the oil without stirring. The rise in temperature is very quick, and the highest point is noted. The difference between the highest and the initial temperature constitutes the heat-of-bromination number.

This number, in the case of Gill and Hatch's calorimeter, is somewhat lower than when a vacuum jacketed tube is employed, and differs somewhat with the diluent of the oil and bromine. In spite of these variations and that due to the personal equation, concordant results may be obtained with the various oils, when the method is carried out under precisely the same conditions. The analyst should carefully work out the test several times with a particular oil till the results agree, and should then with equal care determine the iodine number of the same oil. The iodine number, divided by the heat-of-bromination number, gives the factor which is to be employed under the same conditions for calculating one constant from the other. In the case of Hehner and Mitchell's work with the vacuum tube, measuring 1 cc. of undiluted bromine into 1 gram of oil dissolved in 10 cc. of chloroform, it was found that the factor to be used in calculating the iodine number was 5.5.

The following are some of the results on edible oils obtained by Hehner and Mitchell:

Oil.	Heat of Bromination.	Iodine Number.	Calculated Iodine Number.

solution of 7½ grams of camphor in 25 cc. of carbon tetrachloride, an average rise in temperature of 4.2° was obtained, and the specific temperature reaction is calculated for each oil by dividing the heat of bromination by this number. Furthermore, by dividing the iodine number of several oils by this specific temperature reaction, the factor to be employed for the calculation of the iodine number was found to be 17.18, as in the following cases:*

Oil.	Specific Temperature Reaction.	Iodine Number.	
		Calculated.	Found.
Prime lard	3.705	63.8	63.8
No. 1 lard	4.096	70.3	73.9
Olive	4.762	81.8	82.0
Cottonseed	5.667	97.3	103.0
Corn	6.381	109.5	107.8

THE REFRACTOMETER.—Various forms of refractometer are used in fat and oil analysis. Thus the *Abbé* refractometer, more fully described on page 397, requires only one or two drops of the oil to be examined, and reads the index of refraction directly, using ordinary, white light.

The *Pulfrich* is used with the sodium light, and requires a larger amount of oil than the Abbé, the oil or melted fat being held in a cylinder above the prism. The scale reading is in angular degrees, from which the index of refraction is calculated by a formula or from a table. The instrument is provided with a temperature-controlling apparatus.

In the *Amagat and Jean* or *oleo-refractometer*, an outer and an inner cylinder are respectively filled with an oil of known value or purity, and with the oil to be examined. By the comparative displacement to the right or left of a beam of white light passing through both cylinders, the displacement being read in degrees on an arbitrary scale, the refraction of an oil may be measured. Two oils may thus be readily compared under the same conditions, one of known purity, for example, with a doubtful sample of the same kind.

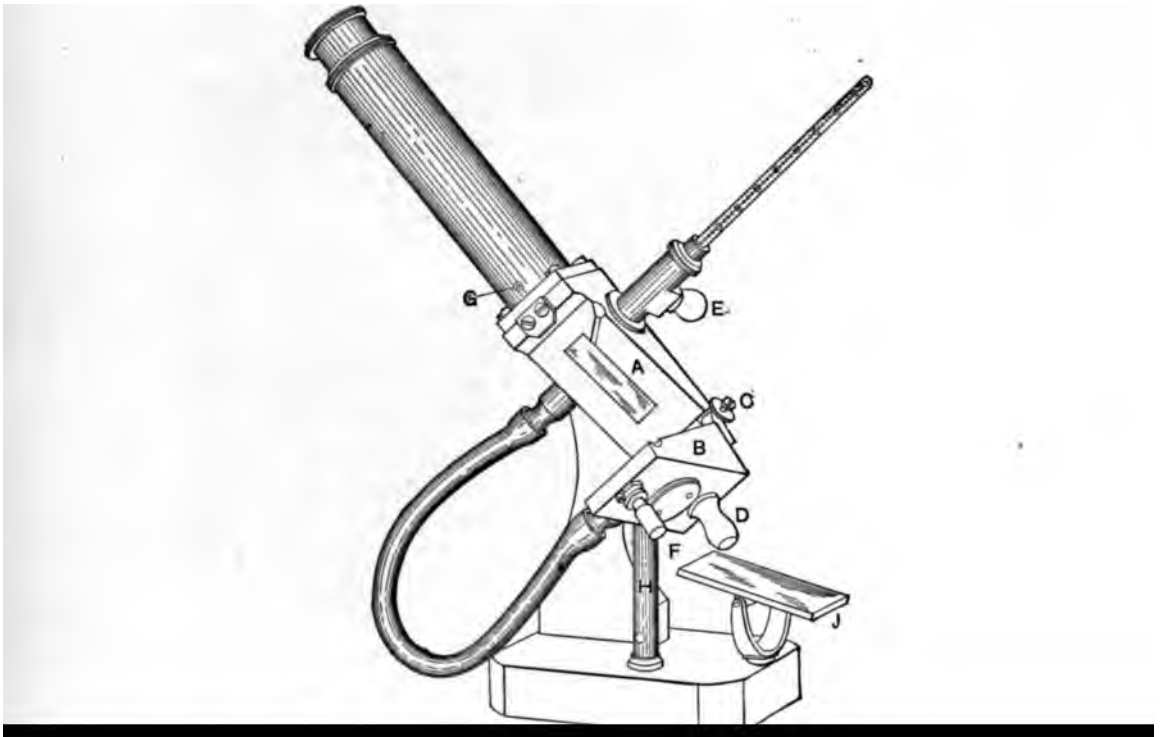
The *butyro-refractometer* and the *Wollny milk fat refractometer* are, as their names imply, instruments primarily intended for more restricted fields of work than the foregoing. They involve the same principle as the Abbé, but are simpler in construction and have arbitrary scales.

Unless such widely varying substances as the waxes and the essential oils are to be studied, the Zeiss butyro-refractometer, though primarily

* Gill, Oil Analysis, p. 128.

intended for the examination of butter and lard, is by far the most useful and convenient form of instrument for the food laboratory, being equally well adapted for examining all the common edible oils and fats.

The Zeiss Butyro-refractometer.—This instrument (shown in Fig. 91) is so constructed that the degree of refraction of a beam of light, which passes obliquely through a thin film of the fat, is read on an arbi-



The refractometer is connected to any kind of heating arrangement, which admits of warm water being transmitted through the prism casing, in at *D* and out at *E*. A simple arrangement, which suffices for all ordinary cases, may expeditiously be improvised in the following manner: Fill a vessel of say 2 gallons capacity with water of 40° to 50° C. Into this vessel dip the free end of an india-rubber tube slipped over the nozzle *D* and let the vessel be placed at a height of about one-half or one yard above the refactometer. Then it will be seen that suction at a tube attached to *E* will cause the warm water to flow through the prism casing by the action of the siphon arrangement. By means of a pinch clip the velocity of the water may be regulated at will. The waste water may be allowed to flow into a second vessel and, provided its temperature does not fall below 30°, it may be used for replenishing the upper vessel.

When working with solid fats, a temperature must be maintained by the heated water well above the melting-point of the fat. With liquid oils no heater is necessary, as determinations may be made at room temperature, but it is advisable in all cases to have a constant stream of water passing through the water jacket, which may be done by directly connecting it with the water faucet in the case of oils, since, without such precautions to insure even temperature, disturbing variations are liable to occur, due to the warming of the prisms by the manipulation of cleaning, etc.

Refractometer Heater.—A regular heater, shown in Fig. 92, is furnished by the manufacturers, capable of supplying a current of water of approximately constant temperature, and will be found of great convenience when the instrument is to be used constantly, especially with the solid fats.

A supply reservoir *A* is secured to the wall and is connected by means of a rubber inlet tube *G* to the water faucet *C*. The reservoir is provided with a waste overflow pipe and with an outlet tube *D*, the flow through the latter being regulated by the cock *H*. The tube *D* leads to the spiral heater *HS*, which is heated by a Bunsen burner. From the heater the tube *E* conducts the warm water through the refractometer, from which it flows through the tube *F*, either directly into the sink, or into the intermediate vessel *B*. The temperature of the water is regulated by adjusting the cock *H*, and the height of the flame of the Bunsen burner.

Manipulation of the Butyro-refractometer.—The prism casing is first opened by giving about half a turn to the right to the pin *F*, Fig. 91, until it meets with a stop. Then simply turn the half *B* of the prism

casing aside. Pillar *H* holds *B* in the position shown in Fig. 91. The prism and metallic surfaces must now be cleaned with the greatest care, the best means for this purpose being soft linen, moistened with a little alcohol or benzine.

If the sample to be examined is a solid fat, maintain the temperature above the melting-point, and apply by a glass rod a drop or two of the clear melted fat (filtered if turbid) to the surface of the prism contained in the casing *B*. For this purpose the apparatus should be raised with

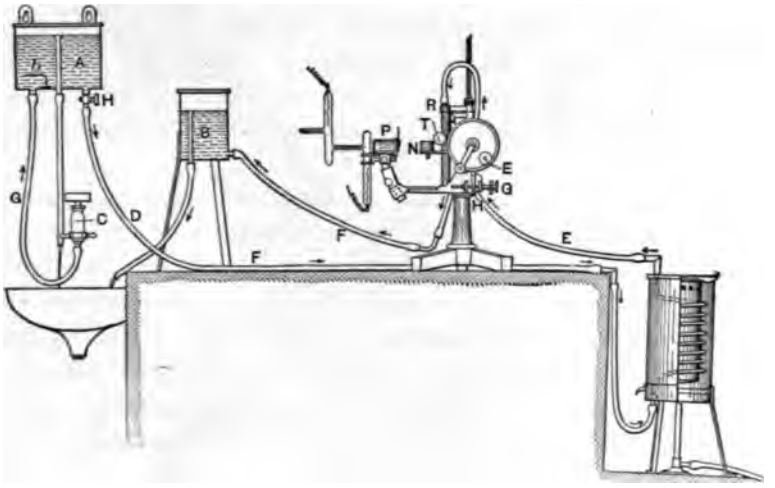


FIG. 92.—The Zeiss Heating Apparatus for all Forms of Refractometer. Shown in the cut in connection with the Pulfrich refractometer.

the left hand so as to place the prism surface in a horizontal position. A liquid oil is directly applied in the same manner without preliminary precautions as to heating. Now press *B* against *A*, and place *E* by

naked eye, placing the latter at its proper distance from the ocular. Finally adjust the movable front part of the ocular so that the scale becomes clearly visible.

By allowing a current of water of constant temperature to flow through the apparatus some time previous to the taking of the reading, the at first somewhat hazy critical line approaches in a short time, generally after a minute, a fixed position, and quickly attains its greatest distinctness. When this point has been reached, note the appearance of the critical line (i.e., whether colorless or colored, and in the latter case of what color); also note the position of the critical line on the centesimal scale, which admits of the tenth divisions being conveniently estimated; at the same time read the position of the thermometer.

Testing the Adjustment of the Ocular Scale.—It is imperative that the adjustment of the instrument be tested periodically, and in particular when it is being used for the first time. This may be done by means of the standard fluid supplied with the instrument, the critical line of which is approximately colorless, and must occupy the following positions in the scale.

Temperature.	Scale Division.	Temperature.	Scale Division.	Temperature.	Scale Division.	Temperature.	Scale Division.
30°	68.1	25°	71.2	20°	74.3	15°	77.3
29°	68.7	24°	71.8	19°	74.9	14°	77.9
28°	69.3	23°	72.4	18°	75.5	13°	78.6
27°	70.0	22°	73.0	17°	76.1	12°	79.2
26°	70.6	21°	73.6	16°	76.7	11°	79.8
25°	71.2	20°	74.3	15°	77.3	10°	80.4

The fractional parts of a degree can accordingly easily be brought into calculation (0.1 = 0.06 scale div.). Deviations of 1 to 2 decimals of the scale divisions are of no consequence, and are in most cases due to inexact determinations of temperature. Should, however, careful tests result in the discovery of greater deviations, readjustment of the scale will be necessary, which may be effected by means of a watch-key supplied with the instrument, in accordance with the values given in the above table. The watch-key is inserted at *G* in Fig. 91, and by its means the position of the objective, and therefore that of the critical line with respect to the scale may be altered.

Transformation of Scale Divisions into Indices of Refraction.—The following table, adapted from that of Pulfrich, enables one to convert scale readings on the butyro-refractometer into indices of refraction, n_D, and vice versa:

EQUIVALENTS OF INDICES OF REFRACTION AND BUTYRO-REFRACTOMETER READINGS.

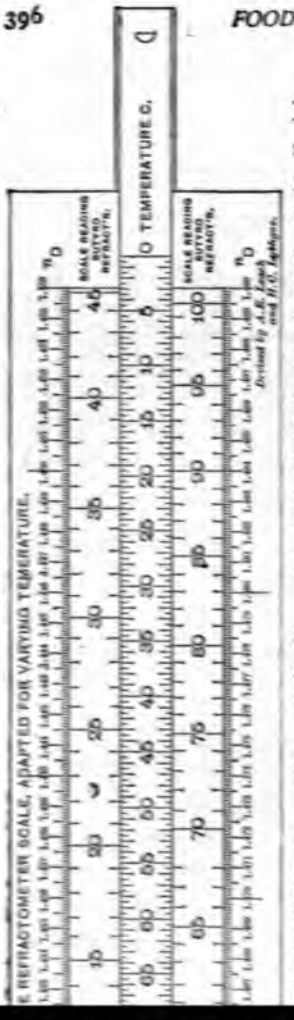
Refractive Index. n_D.	Fourth Decimal of n_D.									
	0	1	2	3	4	5	6	7	8	9
	SCALE READINGS.									
1.422	0.0	0.1	0.2	0.4	0.5	0.6	0.7	0.9	1.0	1.1
1.423	1.2	1.4	1.5	1.6	1.7	1.9	2.0	2.1	2.2	2.4
1.424	2.5	2.6	2.7	2.8	3.0	3.1	3.2	3.3	3.5	3.6
1.425	3.7	3.8	4.0	4.1	4.2	4.3	4.5	4.6	4.7	4.8
1.426	5.0	5.1	5.2	5.4	5.5	5.6	5.7	5.9	6.0	6.1
1.427	6.2	6.4	6.5	6.6	6.8	6.9	7.0	7.1	7.2	7.4
1.428	7.5	7.6	7.7	7.9	8.0	8.1	8.2	8.4	8.5	8.6
1.429	8.7	8.9	9.0	9.1	9.2	9.4	9.5	9.6	9.8	9.9
1.430	10.0	10.1	10.3	10.4	10.5	10.6	10.7	10.9	11.0	11.1
1.431	11.3	11.4	11.5	11.6	11.8	11.9	12.0	12.2	12.3	12.4
1.432	12.5	12.7	12.8	12.9	13.0	13.2	13.3	13.5	13.6	13.7
1.433	13.8	14.0	14.1	14.2	14.4	14.5	14.6	14.7	14.9	15.0
1.434	15.1	15.3	15.4	15.5	15.6	15.8	15.9	16.0	16.2	16.3
1.435	16.4	16.6	16.7	16.8	17.0	17.1	17.2	17.4	17.5	17.6
1.436	17.8	17.9	18.0	18.2	18.3	18.4	18.5	18.7	18.8	18.9
1.437	19.1	19.2	19.3	19.5	19.6	19.7	19.8	20.0	20.1	20.3
1.438	20.4	20.5	20.6	20.8	20.9	21.1	21.2	21.3	21.4	21.6
1.439	21.7	21.8	22.0	22.1	22.2	22.4	22.5	22.6	22.7	22.9
1.440	23.0	23.2	23.3	23.4	23.5	23.7	23.8	23.9	24.1	24.2
1.441	24.3	24.5	24.6	24.7	24.8	25.0	25.1	25.2	25.4	25.5
1.442	25.6	25.8	25.9	26.1	26.2	26.3	26.5	26.6	26.7	26.9
1.443	27.0	27.1	27.3	27.4	27.5	27.7	27.8	27.9	28.1	28.2
1.444	28.3	28.5	28.6	28.7	28.9	29.0	29.2	29.3	29.4	29.6
1.445	29.7	29.9	30.0	30.1	30.3	30.4	30.6	30.7	30.8	30.9
1.446	31.1	31.2	31.4	31.5	31.6	31.8	31.9	32.1	32.2	32.3
1.447	32.5	32.6	32.8	32.9	33.0	33.2	33.3	33.5	33.6	33.7
1.448	33.9	34.0	34.2	34.3	34.4	34.6	34.7	34.9	35.0	35.1
1.449	35.3	35.4	35.6	35.7	35.8	36.0	36.1	36.3	36.4	36.5
1.450	36.7	36.8	37.0	37.1	37.2	37.4	37.5	37.7	37.8	37.9
1.451	38.1	38.2	38.3	38.5	38.6	38.7	38.9	39.0	39.2	39.3
1.452	39.5	39.6	39.7	39.9	40.0	40.1	40.3	40.4	40.6	40.7
1.453	40.9	41.0	41.1	41.3	41.4	41.5	41.7	41.8	42.0	42.1

EQUIVALENTS OF INDICES OF REFRACTION AND BUTYRO-REFRACTOMETER READINGS—(*Continued*).

Refractive Index. n_D.	Fourth Decimal of n_D.									
	0	1	2	3	4	5	6	7	8	9
	SCALE READINGS.									
1.470	66.4	66.5	66.7	66.8	67.0	67.2	67.3	67.5	67.7	67.8
1.471	68.0	68.1	68.3	68.4	68.6	68.7	68.9	69.1	69.2	69.4
1.472	69.5	69.7	69.9	70.0	70.2	70.3	70.5	70.7	70.8	71.0
1.473	71.1	71.3	71.4	71.6	71.8	71.9	72.1	72.2	72.4	72.5
1.474	72.7	72.9	73.0	73.2	73.3	73.5	73.7	73.8	74.0	74.1
1.475	74.3	74.5	74.6	74.8	75.0	75.1	75.3	75.5	75.6	75.8
1.476	76.0	76.1	76.3	76.5	76.7	76.8	77.0	77.2	77.3	77.5
1.477	77.7	77.9	78.1	78.2	78.4	78.6	78.7	78.9	79.1	79.2
1.478	79.4	79.6	79.8	80.0	80.1	80.3	80.5	80.6	80.8	81.0
1.479	81.2	81.3	81.5	81.7	81.9	82.0	82.2	82.4	82.5	82.7
1.480	82.9	83.1	83.2	83.4	83.6	83.8	83.9	84.1	84.3	84.5
1.481	84.6	84.8	85.0	85.2	85.3	85.5	85.7	85.9	86.0	86.2
1.482	86.4	86.6	86.7	86.9	87.1	87.3	87.5	87.6	87.8	88.0
1.483	88.2	88.3	88.5	88.7	88.9	89.1	89.2	89.4	89.6	89.8
1.484	90.0	90.2	90.3	90.5	90.7	90.9	91.1	91.2	91.4	91.6
1.485	91.8	92.0	92.1	92.3	92.5	92.7	92.9	93.0	93.2	93.4
1.486	93.6	93.8	94.0	94.1	94.3	94.5	94.7	94.8	95.0	95.2
1.487	95.4	95.6	95.8	96.0	96.1	96.3	96.6	96.7	96.9	97.0
1.488	97.2	97.4	97.6	97.8	98.0	98.1	98.3	98.5	98.7	98.9
1.489	99.1	99.2	99.4	99.6	99.8	100.0				

The Critical Line.—It should be remembered that the instrument is primarily intended for use with butter, and that the prisms are so constructed that the critical line of pure butter is colorless, while various other fats and oils, notably oleomargarine, which have greater dispersive powers than natural butter, show a colored critical line. When too great dispersion is apparent to render possible an accurate reading, or when the critical line presents very broad fringes, as with linseed oil, poppyseed oil, turpentine, etc., use a sodium light, obtained by the application of table salt to the Bunsen gas flame, or the diffused daylight may be reflected in the mirror through a flat bottle filled with a dilute solution of potassium bichromate, to give a yellow light.

The advantages of the refractometer for examination of fats and oils consist in the convenience with which very accurate determinations of the refractive index may be made at any temperature between 10° and 50° C., inclusive of thermal variations of refractive powers, and also in the possibility which it affords of distinguishing substances by their different dispersive powers, rendered visible by the different coloring of the critical line, a red-colored critical line being indicative of a relatively low dispersive power, a blue line of relatively high dispersion.



Variation of Reading with the Temperature.—No definite temperature has been adopted as a standard for readings of this instrument, but it is easy to reduce readings at any temperature to terms of any other temperature for purposes of comparison. While the change in index of refraction for 1° C. is the same whatever the temperature, as Tolman and Munson have pointed out,* the change in scale reading per 1° C. decreases as the temperature increases, and varies slightly with different oils. For correcting reading R' at a temperature T' to a reading R at temperature T, their formula is $R=R'-X(T-T')$, X being the change in scale reading due to change of 1° C. in temperature.

For butter, oleomargarine, beef tallow, lard, and other fats reading from 40° to 50° or thereabouts on the scale, $X=0.55$. For oils reading between 60° and 70°, like olive, mustard, rapeseed, cottonseed, peanut, etc., $X=0.58$, and for oils reading between 70° and 80°, like corn oil, $X=0.62$.

The slide rule † shown in Fig. 93, for use with the refractometer, has been jointly devised by H. C. Lythgoe and the writer, to render unnecessary the use of tables or formulas. The extreme upper and lower scale divisions indicate indices of refraction, and adjacent to these are the scale

in comparing the work of various observers, where different temperatures have been employed.

The correction for change in n_D on the scale is 0.000365 for 1° C., being based on the experimental work of Tolman, Long, Proctor, Lythgoe, and the author.

The table on pp. 398, 399 is useful as showing readings on the butyro-refractometer of all the edible oils and fats at various temperatures.*

The Abbé Refractometer, Fig. 94, has a much wider range in reading

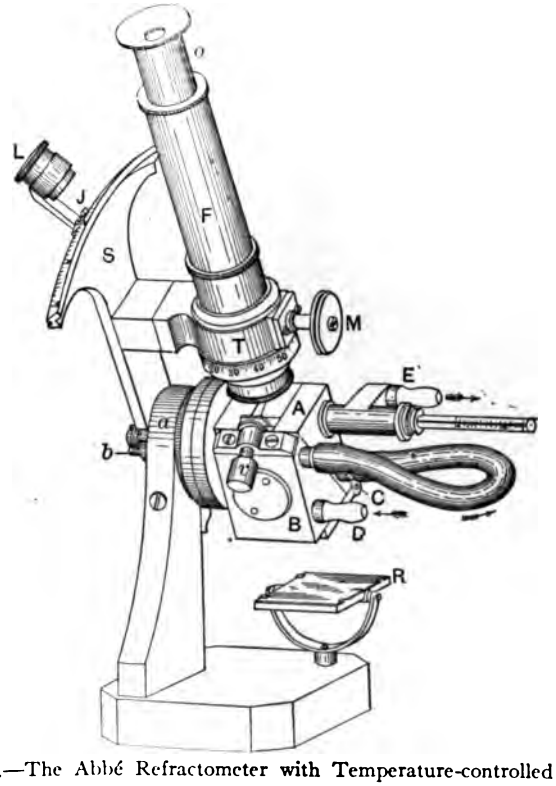


FIG. 94.—The Abbé Refractometer with Temperature-controlled Prisms.

than either the butyro or the Wollny instruments already described, reading as it does to the fourth decimal between the limits of 1.3 and 1.7 in indices of refraction. The equivalent readings of the Wollny milk fat refractometer, in indices of refraction, range from 1.3332 to 1.4220, while those of the butyro-instrument run from 1.4220 to 1.4895. The Abbé instrument is thus necessary for use with the high-refracting essential

* Lythgoe, Tech. Quarterly.

CALCULATED READINGS ON BUTYRO-REFRACTOMETER OF EDIBLE OILS AND FATS.

Temp. C.	Cocoanut Oil.	Butter.*	Beef Stearin.	Cacao Butter.	Beef Tallow.	Lard Stearin.	Beef Oleo.	Lard.†	Lard Oil.
45.0	31.6	41.5	41.9	43.7	44.1	44.9	45.0	48.2	
44.5	31.9	41.8	42.2	44.0	44.3	45.1	45.3	48.4	
44.0	32.2	42.0	42.4	44.2	44.6	45.5	45.6	48.7	
43.5	32.4	42.3	42.6	44.5	44.8	45.7	45.9	49.0	
43.0	32.7	42.6	42.9	44.8	45.1	46.0	46.1	49.3	
42.5	52.9	42.9	43.2	45.0	45.4	46.3	46.4	49.6	
42.0	33.2	43.1	43.5	45.3	45.6	46.5	46.7	49.9	
41.5	33.5	43.4	43.7	45.6	45.8	46.8	47.0	50.1	
41.0	33.7	43.7	44.0	45.9	46.1	47.0	47.3	50.4	
40.5	34.0	43.9	44.2	46.1	46.3	47.3	47.6	50.7	
40.0	34.3	44.2	44.5	46.4	46.6	47.6	47.8	51.0	51.6
39.5	34.5	44.5	.....	46.6	46.8	47.8	48.1	51.3	51.8
39.0	34.8	44.8	.....	46.8	47.1	48.1	48.4	51.6	52.1
38.5	35.0	45.0	.....	47.1	47.4	48.4	48.7	51.9	52.4
38.0	35.3	45.3	.....	47.4	47.6	48.6	48.9	52.1	52.6
37.5	35.5	45.6	.....	47.6	47.8	48.9	49.2	52.4	52.8
37.0	35.8	45.9	.....	47.9	48.1	49.2	49.5	52.7	53.1
36.5	36.1	46.1	.....	48.2	48.3	49.4	49.8	53.0	53.4
36.0	36.3	46.4	.....	48.5	48.6	49.7	50.0	53.3	53.7
35.5	36.6	46.7	.....	48.7	48.8	50.0	50.3	53.6	54.0
35.0	36.9	47.0	.....	49.0	49.1	50.2	50.6	53.9	54.2
34.5	37.1	47.2	.....	.....	.....	.....	50.9	54.2	54.5
34.0	37.4	47.5	.....	.....	.....	.....	51.2	54.4	54.7
33.5	37.6	47.8	.....	.....	.....	.....	51.5	54.7	55.0
33.0	37.9	48.1	.....	.....	.....	.....	51.7	55.0	55.3
32.5	38.1	48.3	.....	.....	.....	.....	52.0	55.3	55.6
32.0	38.4	48.6	.....	.....	.....	.....	52.3	55.6	55.9
31.5	38.6	48.9	.....	.....	.....	.....	52.6	55.9	56.1
31.0	38.9	49.2	.....	.....	.....	.....	52.8	56.1	56.4
30.5	39.2	49.5	.....	.....	.....	.....	53.1	56.4	56.7

[redacted]

CALCULATED READINGS—(*Continued*).

Temp. C.	Olive Oil.	Peanut Oil.	Cotton-seed Oil.	Rape-seed Oil.	Sesame Oil.	Yellow Mustard Oil.	Black Mustard Oil.	Sun-flower Oil.	Corn Oil.	Poppy-seed Oil.
35.0	57.0	59.8	61.8	62.1	62.3	63.0	64.2	64.5	65.0	65.5
34.5	57.2	60.0	62.1	62.4	62.5	63.3	64.5	64.8	65.3	65.8
34.0	57.4	60.3	62.3	62.7	62.8	63.6	64.8	65.1	65.6	66.1
33.5	57.7	60.6	62.5	63.0	63.1	63.9	65.1	65.4	65.9	66.4
33.0	58.0	60.9	62.8	63.3	63.4	64.1	65.3	65.7	66.2	66.7
32.5	58.3	61.1	63.0	63.6	63.7	64.4	65.6	66.0	66.5	67.0
32.0	58.5	61.4	63.2	63.8	64.0	64.7	65.9	66.3	66.8	67.3
31.5	59.0	61.7	63.6	64.1	64.3	65.0	66.2	66.6	67.1	67.6
31.0	59.2	62.0	64.0	64.4	64.6	65.3	66.5	66.9	67.4	67.9
30.5	59.5	62.2	64.2	64.7	64.9	65.6	66.8	67.2	67.7	68.2
30.0	59.9	62.5	64.5	65.0	65.1	65.8	67.0	67.5	68.0	68.5
29.5	60.1	62.8	64.9	65.3	65.4	66.1	67.3	47.7	68.2	68.7
29.0	60.3	63.1	65.1	65.6	65.7	66.4	67.6	68.0	68.5	69.0
28.5	60.6	63.3	65.3	65.9	66.0	66.7	67.9	68.3	68.8	69.3
28.0	60.9	63.6	65.7	66.1	66.2	66.9	68.1	68.6	69.1	69.6
27.5	61.1	63.9	66.0	66.4	66.5	67.2	68.4	68.9	69.4	69.9
27.0	61.5	64.2	66.5	66.7	66.8	67.5	68.7	69.2	69.7	70.2
26.5	62.0	64.4	67.0	67.0	67.1	67.8	69.0	69.5	70.0	70.5
26.0	62.2	64.7	67.3	67.3	67.0	68.0	69.2	69.8	70.3	70.8
25.5	62.4	65.0	67.5	67.6	67.7	68.3	69.5	70.1	70.6	71.1
25.0	63.0	65.3	67.9	67.8	67.9	68.6	69.8	70.4	70.9	71.4
24.5	63.3	65.5	68.2	68.1	68.2	68.9	70.1	70.7	71.2	71.7
24.0	63.6	65.8	68.5	68.4	68.5	69.2	70.4	71.0	71.5	72.0
23.5	63.9	66.1	68.8	68.7	68.8	69.5	70.7	71.3	71.8	72.3
23.0	64.2	66.4	69.1	69.0	69.1	69.7	70.9	71.6	72.1	72.6
22.5	64.5	66.6	69.4	69.3	69.4	70.0	71.2	71.9	72.4	72.9
22.0	64.8	66.9	69.7	69.7	69.7	70.3	71.5	72.2	72.7	73.2
21.5	65.1	67.1	70.0	70.0	70.0	70.6	71.8	72.5	73.0	73.5
21.0	65.4	67.4	70.3	70.3	70.3	70.9	72.1	72.8	73.3	73.8
20.5	65.7	67.7	70.6	70.6	70.5	71.2	72.4	73.1	73.6	74.1
20.0	66.0	68.0	70.9	70.8	70.8	71.4	72.6	73.4	73.9	74.4
19.5	66.3	68.2	71.2	71.1	71.1	71.7	72.9	73.6	74.1	74.6
19.0	66.6	68.5	71.5	71.4	71.4	72.0	73.2	73.9	74.4	74.9
18.5	66.9	68.8	71.8	71.7	71.7	72.3	73.5	74.2	74.7	75.2
18.0	67.2	69.1	72.1	72.0	72.0	72.6	73.8	74.5	75.0	75.5
17.5	67.5	69.3	72.4	72.3	72.3	72.9	74.1	74.8	75.3	75.8
17.0	67.8	69.6	72.7	72.6	72.5	73.1	74.3	75.1	75.6	76.1
16.5	68.1	69.9	73.0	72.9	72.8	73.4	74.6	75.4	75.9	76.4
16.0	68.4	70.2	73.3	73.2	73.1	73.7	74.9	75.7	76.2	76.7
15.5	68.7	70.5	73.6	73.5	73.4	74.0	75.2	76.0	76.5	77.0
15.0	68.9	70.8	73.8	73.8	73.7	74.3	75.5	76.3	76.8	77.3

oils. Its construction is such that the prisms can withstand a higher heat than in the case of the butyro-refractometer, and it is hence better adapted for the examination of samples having a high melting-point, such as beeswax and paraffin. An advantage of the Abbé over the butyro instrument lies in the fact that the wide dispersion, inevitable when read ing many of the oils on the butyro, may be entirely compensated for with the Abbé, and a clear sharp line be obtained. The construction of the prisms in relation to the heating jacket is similar in both instruments, and a film of the oil or fat to be examined is held in the same manner between the surfaces of the prisms.

Looking through the ocular *O* of the Abbé, Fig. 94, the arm *J*, bearing the telescope *L*, is turned back and forth until the line of demarkation of the refracted beam passing through the oil is seen to coincide with the centering cross hairs. By turning the thumb screw *M*, the graduated chamber immediately below containing the compensator may be rotated sufficiently to entirely do away with any dispersion of the oil, thus getting a perfectly clear, sharp reading.

The index of refraction is read through the telescope *L* on the graduated arc *S* to the fourth decimal place.

The Acetyl Value. — On heating fats with acetic anhydride they become "acetylated"; i.e., the hydrogen atom of their alcoholic hydroxyl group is exchanged for the acetic acid radicle, in accordance, for example, with the following reaction:

$$\underset{\text{Ricinoleic acid}}{C_{17}H_{32}OH\cdot COOH} + \underset{\text{Acetic anhydride}}{(C_2H_3O)_2O} = \underset{\text{Acetyl-ricinoleic acid}}{C_{17}H_{32}O\cdot C_2H_3O\cdot COOH} + \underset{\text{Acetic acid}}{C_2H_4O_2}.$$

By the actyl value is meant the number of milligrams of potassium

water, which are in turn siphoned off, the operation being repeated till the wash water tests free from acid by litmus paper.

The acetylated fat is then separated from the water by drying at 100° in an oven.

From 2 to 4 grams of the acetylated fat is weighed into a flask, and saponified with alcoholic potash in precisely the same manner as for the determination of the saponification number. Evaporate the alcohol and dissolve the soap in water. One of two methods may be carried out for freeing the acetic acid for titration, one by distillation and the other by filtration.

For the former or distillation process, acidify the aqueous solution of the soap with 1:10 sulphuric acid, and distill in the same way as in the Reichert process, excepting that in this case from 600 to 700 cc. of distillate must be obtained, so that water should be added from time to time through a stoppered funnel fixed in the cork of the distilling-flask. The distillate should be received in a funnel with a loose cotton plug, so as to filter it free from insoluble acids mechanically carried over. The filtrate is titrated with tenth-normal sodium hydroxide, using phenolphthalein as an indicator. The number of cubic centimeters of alkali used is multiplied by 5.61, and the product divided by the number of grams of acetylated fat taken. The result is the acetyl value.

If the filtration process is used (which is more rapid and should give concordant results with the distillation process), the exact amount of alcoholic potash used in the saponification should be accurately measured in carrying out the former part of the test, and the exact number of cubic centimeters of standard acid corresponding to the amount of alkali employed should be added to the aqueous soap solution. The mixture should be gently warmed, and the fatty acids will gather in a layer at the top. These are filtered off and washed, till free from acid, with boiling water. The filtrate is titrated with tenth-normal sodium hydroxide, and the acetyl value calculated as in the distillation process.

EDIBLE OILS ARRANGED IN ORDER OF ACETYL VALUE.

	Average.
Cottonseed oil	18.0
Rape oil	14.7
Poppyseed oil	13.1
Sesame oil	11.5
Olive oil	10.6
Peanut oil	3.4

The Valenta Test.—This depends upon the solubility of the oil in glacial acetic acid. Pour from 3 to 5 cc. of the oil into a test-tube, and add an equal volume of glacial acetic acid (specific gravity 1.0562). Place a thermometer in the tube and warm gently till the oil goes into solution. Then allow the mixture to cool, and observe the temperature at which the solution begins to appear turbid.

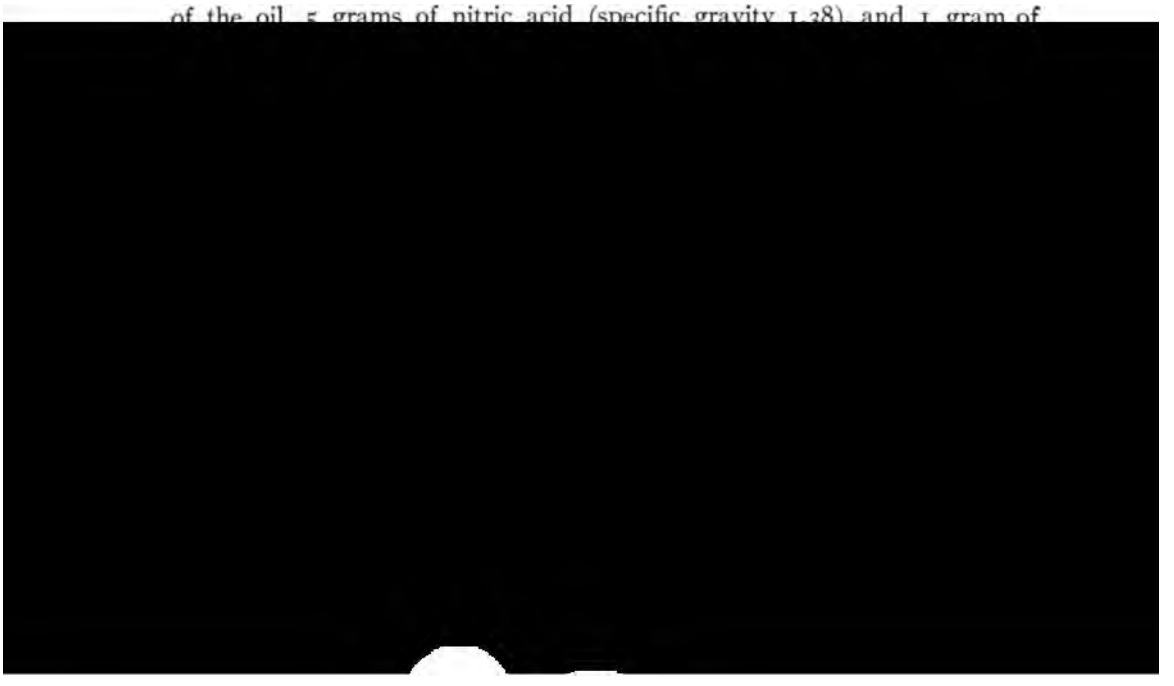
Castor oil and oil of the olive kernel are soluble in glacial acetic acid at ordinary temperatures, while rape and mustard seed oils are insoluble even in the boiling acid.

Elaïdin Test.—This is based on the conversion by nitrous oxide of liquid olein into the solid elaïdin, a crystalline compound isomeric with olein, while other common glycerides remain liquid under treatment with this reagent. By the consistency of the final product, when subjected under certain conditions to the action of nitrous oxide, some idea as to the character of the oil may be gained.

Manipulation.—To carry out the test according to Pontet (modified), weigh 5 grams of the oil into a beaker, add 7 grams of nitric acid (specific gravity 1.34) and about 0.5 gram of copper wire. Place the beaker in water at 15° and stir thoroughly with a glass rod in such a manner as to make an intimate mixture of the oil and the evolved nitrous oxide gas. After the wire has been dissolved, add another piece of about the same size and again stir vigorously. Set aside for about two hours, at the end of which, in the case of pure olive, almond, peanut, or lard oil, it will have been changed into a solid white mass.

Nearly all the seed oils, especially cottonseed and mustard, are turned into a pasty or buttery mass.

Another modification of Pontet's test consists in mixing 10 grams of the oil, 5 grams of nitric acid (specific gravity 1.38), and 1 gram of



been carefully neutralized with a weak solution of sodium hydroxide, using phenolphthalein as an indicator. Warm the mixture to about 60°, and add carefully from a burette tenth-normal sodium hydroxide (using the above indicator) till a pink color is produced, shaking thoroughly during the titration.

The result may be reported in terms of percentage of oleic acid (each cubic centimeter of tenth-normal alkali is equivalent to 0.0282 gram of oleic acid) or as the "acid number," by which is meant the number of cubic centimeters of tenth-normal alkali necessary to saturate the free acid in 1 gram of the fat or oil.

Constants of the Free Fatty Acids.—Often much information as to the character of an oil or fat may be obtained by determining such constants of its fatty acids as the melting- and solidifying-point, the iodine number, etc.

To prepare the fatty acids for examination, saponify a quantity of the oil or fat with alcoholic potash, evaporate the alcohol, and dissolve the soap in hot water. Decompose the soap by the addition of an excess of hydrochloric or sulphuric acid, continuing the heating till the fatty acids rise in a layer to the top of the liquid, from which they may be removed. The melting-point, iodine number, etc., are determined as with the oil or fat itself.

Solidifying-point of the Fatty Acids, or Titer Test.—*Method of Dalican.**—Liberate the fatty acids as in the preceding sections, free from water by filtration, and allow to stand over night in a porcelain dish in a desiccator. The mass of fatty acids is then melted and poured into a warm test-tube 3.5 cm. in diameter till it is about half full. The test-tube is then inserted into the neck of a flask, either directly or, preferably, is fitted into a rubber stopper, which itself fits the flask. A delicate thermometer previously warmed is then inserted into the liquid, and the contents are stirred with it, taking care that it does not touch the sides, but is raised and lowered so that the whole is well mixed as portions of the mass solidify. The temperature at first falls, and the thermometer should be watched carefully, for, after a short time, it quickly rises a few tenths of a degree and remains stationary for a minute or two before again falling. This stationary point is known as the solidifying-point or titer.

UNSAPONIFIABLE MATTER.—As will be seen by reference to the table on page 411, the unsaponifiable matter in pure edible oils and fats is

* Thorpe's Dictionary of Applied Chemistry, vol. III, p. 50.

comparatively insignificant in amount, consisting largely of cholesterol or phytosterol. A high content of unsaponifiable matter is indicative of adulteration, pointing to the presence of mineral or coal-tar oils, or to paraffin.

Determination of Unsaponifiable Matter.*—Weigh 7 to 10 grams of the fat or oil in a 250-cc. flask, and saponify by boiling with 25 cc. of alcoholic potassium hydroxide and 25 cc. of alcohol under a return-flow condenser. After saponification, add 30 to 40 cc. of water, and bring to the boiling-point. Cool and transfer the contents from the flask to a separatory funnel, washing out the flask first with a small amount of 50% alcohol, and finally with 50 cc. of petroleum ether (B.P. 40°–70°), adding both washings to the separatory funnel. Shake the latter thoroughly, but avoid if possible forming an emulsion. If the latter persists in forming, add a volume of water equal to that of the soap solution, which will sometimes break it up. After separation of the petroleum ether layer, draw off the underlying soap solution into a beaker, and wash the petroleum ether two or three times with 50% alcohol, which is drawn off and added to the soap solution. The petroleum ether is then run into a tared Erlenmeyer flask, and the soap solution extracted twice more with fresh portions of petroleum ether, washing the ether each time with 50% alcohol as before and then transferring the ether to the tared flask. The petroleum ether is then removed by placing the flask on the water-bath, bumping being prevented by means of a spiral of platinum wire weighed with the flask. Finally remove all traces of remaining ether by blowing hot air through the flask, or, in the absence of mineral oils (some of which are volatile), dry in the water-oven to constant weight, cool in a desiccator, and weigh.

CHOLESTEROL AND PHYTOSTEROL.—These are monatomic alcohols

soluble in cold, but readily soluble in boiling alcohol, and soluble in ether, chloroform, methyl alcohol, benzene, and oil of turpentine. It sublimes unchanged at 200°, but at higher temperatures decomposes.

Commercial cholesterol is obtained from wool oil and is known as lanolin, being used largely in medicine as a basis for ointment.

Cholesterol occurs also in the yolk of eggs, in many animal secretions, and in most animal oils and fats.

It separates in laminated, transparent crystals from a mixture of 2 volumes alcohol and 1 volume ether, and in the form of anhydrous needles from chloroform.

Phytosterol ($C_{26}H_{44}O,H_2O$) is most abundantly found in the leguminous seeds, and is prepared commercially from these, especially from peas and lentils. It is a constituent of most vegetable oils.

It crystallizes in slender, glittering plates from chloroform, ether, and petroleum ether, and from alcohol in tufts of needles. In solubility it much resembles cholesterol, but its melting-point from 132° to 134° is lower.

Separation of Cholesterol and Phytosterol from Oils and Fats.—*Method of Kreis and Wolf.**—50 grams of fat are boiled with 125 cc. of 95% alcohol and 25 cc. of a 40% aqueous solution of sodium hydroxide till saponified. Evaporate off the alcohol, and dissolve the soap in 500 cc. of boiling water, neutralizing the excess of alkali with hydrochloric acid (specific gravity 1.124). Add 100 cc. of a 10% solution of calcium chloride, and shake the flask till the calcium soap separates out, adding more calcium chloride if the liquid still froths after shaking. The calcium soap is collected on a filter of cotton cloth and pressed between filter-paper, after which it is boiled for an hour in 100 cc. of 95% alcohol. When cold, the solution is filtered, and the filtrate treated with 3 cc. of the 40% soda solution to saponify any remaining fat, and then evaporated to dryness. The small residue is powdered and shaken in a flask with 50 cc. of ether at intervals during an hour. The residue obtained on filtering and evaporating the ether is dissolved in as small as possible a volume of hot alcohol, and in cooling the cholesterol or phytosterol is deposited in firm, white crystals.

Determination of Cholesterol and Phytosterol.—*Method of Forster and Reichmann.*†—50 grams of the oil or fat are boiled for five minutes in a flask connected with a reflux condenser with two successive portions

* Chem. Zeit., 1898 (22), p. 805; Jour. Soc. Chem. Ind., 17, p. 1075.

† Analyst, 1897 (22), 131.

of 75 cc. of 95% alcohol, and in each case the alcoholic solution is separated by means of a separatory funnel. The combined alcoholic solutions are then boiled in a flask provided with a funnel in the neck, till one-fourth of the alcohol is evaporated, and then poured into an evaporating dish and brought to dryness. The residue is then extracted with ether, and the ether solution is evaporated to dryness, taken up again with ether, filtered, evaporated once more, and dissolved in hot 95% alcohol, from which it is allowed to crystallize. Cholesterol or phytosterol will crystallize out under these conditions, and may be weighed.

Distinguishing between Cholesterol and Phytosterol.—It is sometimes of importance to determine which of these substances is present in an oil, or whether indeed both occur. Confirmatory proof as to the presence of vegetable in animal oils may, for instance, be established by showing whether the unsaponifiable residue in the sample contains cholesterol or phytosterol or both. Hehner* has made use of this test in determining the presence of cottonseed oil in lard.

The most ready means of distinguishing between cholesterol and phytosterol is furnished by the melting-point (cholesterol 149°, phytosterol 132° to 134°) and by the marked difference between the form of the crystals and the manner of crystallization of the two substances.

Crystallization.—Bömer† has shown that if allowed to crystallize very slowly out of a strong alcoholic solution, preferably absolute alcohol, in a shallow glass, the crystallization in the case of cholesterol alone begins from the margin of the liquid and gradually extends inward toward the center, forming a uniformly bright, thin, colorless film over the whole surface. This film is best removed with a knife or spatula and pressed between filter-paper. The film will be seen, even megascopically, to



foreign matter. The first procedure is the same as above described for cholesterol, the crystals being allowed to separate slowly out of a solution in absolute alcohol. Unlike cholesterol, no film is formed on the surface, but needles (sometimes 1 cm. in length) are gradually eliminated, beginning at the margin and extending inward mostly at the bottom. In concentrated solutions, fine needles would be uniformly deposited through the liquid. These are best separated from the mother liquid by filtration, as they are not easily taken out with a knife. They may be washed on the filter with small amounts of absolute alcohol for microscopical examination, or repeatedly recrystallized, as in the case of cholesterol, till the melting-point is constant.

*Cholesterol Crystals.**—When crystallized separately under above conditions, cholesterol crystals viewed under the microscope show generally rhomboidal forms of plates, as in Fig. 95, but sometimes with a reenter-

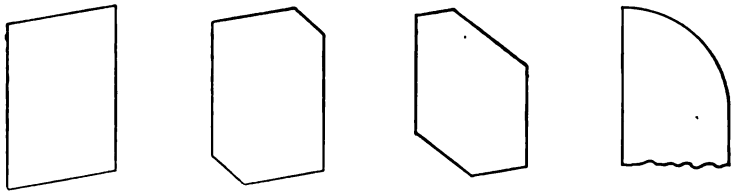


FIG. 95.—Cholesterol Crystals under the Microscope. (After Bömer.)

ing angle. The plates are often grown together in masses. The most characteristic forms are found from the first crystallization or from the first film removed. Sometimes quadrilateral crystals predominate among the plates, often also the other shapes shown are found most numerous.

Phytosterol Crystals.—Pure phytosterol crystallizes in needles or narrow plates, arranged commonly in star form or in bunches. The most common forms are shown in Fig. 96, best conditions as to shape of crystals being obtained from slow crystallization, in which case the needles are finer and more regular.

The crystals are commonly in the form of long, narrow plates, thin and slender, often pointed at both ends. Sometimes the points are lacking, or the ends are beveled. The more frequently they are recrystallized, the larger and more varied are the crystal forms. The broad, hexagonal and quadrilateral plates shown are products of re-

* Zeits. für Nahr. u. Genuss., 1898, pp. 42 and 44.

crystallization; the shorter forms are rarely met with. Sometimes various forms are found side by side in the same crystallization.

Phytosterol crystals, from a second or third recrystallization, sometimes grow together in bunches resembling at first glance to the naked eye the cholesterol masses. They never do this in the first crystallization, whereas in the case of cholesterol the growing together in masses is very characteristic of the first crystallization.

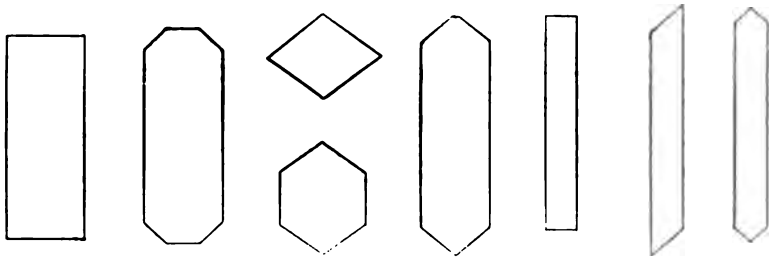


FIG. 96.—Phytosterol Crystals. (After Bömer.)

Thus for purposes of distinguishing between the two the product of the first crystallization is best observed.

Crystals of Mixed Cholesterol and Phytosterol.—In mixtures of the two they do not crystallize separately, but when in nearly equal proportion, or with phytosterol predominating, the crystals much resemble phytosterol. Even when cholesterol predominates to the extent of 20 parts to 1 of phytosterol, the mode of crystallization leans most toward that of phytosterol, though the needles are of different shape. Such a mixture, for instance, does not form in a film like cholesterol, but, like phytosterol, comes out in needle-like bunches. The needles, however, are more often like those shown in Fig. 97 when viewed under the micro-



Paraffin, sometimes present as an adulterant of fats, is best determined as follows:* Boil 2 grams of the fat with 10 cc. of 95% alcohol and 2 cc. of 1:1 sodium hydroxide solution, connect the flask with a reflux condenser, and heat for an hour on the water-bath, or until saponification is complete. Remove the condenser, and allow the flask to remain on the bath till the alcohol is evaporated off and a dry residue is left. Treat the residue with about 40 cc. of water and heat on the bath, with frequent shaking, till everything soluble is in solution. Wash into a separatory funnel, cool, and extract with four successive portions of petroleum ether, which are collected in a tared flask or capsule. Remove the petroleum ether by evaporation and dry in the oven to constant weight.

It should be noted that any phytosterol or cholesterol present in the fat would come down with the paraffin, but the amount would be so insignificant that with added paraffin actually present, it may be disregarded. The character of the final residue should, however, be confirmed by determining its melting-point and specific gravity, and by subjecting it to examination in the butyro-refractometer. The melting-point of paraffin is about 54.5° C.; its specific gravity at 15.5° is from 0.868 to 0.915, and on the butyro-refractometer the reading at 65° C. is from 11 to 14.5.

MICROSCOPICAL EXAMINATION OF OILS AND FATS.

Excepting in the case of solid fats, the use of the microscope has hitherto been comparatively restricted. In the examination of lard and butter for adulterants, the use of the microscope is often of great value, and will be described more fully under these special fats. In general the best fat crystals are obtained by slow crystallization at room temperature from an ether solution, or from a mixture of ether and alcohol. The first crystals formed may often with advantage be filtered out, and washed with the alcohol and ether mixture on the filter, dissolved finally in ether, and the latter allowed to evaporate spontaneously. The crystals are then examined in a medium of ether.

If it be desired to separate the liquid oleins from an oil, so that crystals of the solid fats are left for examination, Gladding† recommends dissolving the fat in a mixture of two volumes of absolute alcohol and one volume of ether in a test-tube, which is stoppered with cotton and set for half an hour in ice water, during which time the more solid stearin

* U. S. Dept. of Agric., Bur. of Chem., Bul. 65, p. 46.

† Jour. Am. Chem. Soc., 1896, 18, p. 189.

MOST COMMON EDIBLE FATS AND OILS AND THEIR ADULTERANTS.

When otherwise stated, the constants are mainly those of Lewkowitsch.

ting-int.	Solidifying-point.	Reichert-Meissl Number.	Saponification Number.	Iodine Number.	Bromine Index.	Insoluble Fatty Acids, Hehner's Number.	Maumené Number.	Specific Temperature Reaction.
.....	−16 to −20	0.0	190.1 to 197	133 to 143	0.835*	94.2 to 95.2	86 to 88	.....
.....	−10	.....	193.5	119 to 135	.....	95	72	.....
.....	.....	4 to 5	188 to 193	113 to 125	.....	93 to 96	81 to 86	6.381†
to 4	.....	0.95	193 to 195	108 to 110	0.645*	95 to 96	75 to 90	1.69 to 170‡
.....	−5	1.2	189 to 193	103 to 108	0.695*	95.7	65.5	.....
.....	−2 to −10	0.6‖	170 to 179	94 to 102	0.632§	95.1	55 to 64	1.25 to 1.44‡
.....	−17	.....	174	96 to 110	0.763§	95.1	43	.....
.....	−8 to −16	.....	170 to 174	92 to 97	.....	96.2	44 to 49	.....
.....	−3 to 0	.....	190 to 196	83 to 100	0.530*	95.8	45 to 51	105 to 137‡
.....	−6 to +2	0.6¶	185 to 196	79 to 88	0.5 to 0.544*	95	41.5 to 45	0.89 to 0.95‡
.....	.....	.....	195 to 196**	52.5† to 85††	.....	.....	41 to 45**	.....
to 32	16 to 22	.....	195	90 to 103	.....	95.9	48	.....
to 33	23 to 21.5	0.2 to 0.8	193.5	32 to 41	.....	94.59	.....	.....
to 24	22 to 14	7 to 8.4	246 to 260	8 to 9.5	.....	88.6 to 90	.....	.....
40.5	27.1 to 29.9	1.10¶	195.4	50 to 70	.....	93 to 96	24 to 27.5	3.71 to 4.09†
to 45	35 to 27	0.5	193.2 to 200	38 to 46	.....	95.6	.....	.....
to 45	36 to 41	.....	192 to 195.2	35 to 46	.....	95.5	.....	.....
to 33	29 to 23	25 to 30.4	227	26 to 38	.....	86.5 to 89.8	.....	.....

Oil or Fat.	Heat of Bromination.	Zeiss Refractometer Reading.	Temperature of Reading.	Acetyl Value.	Free Fatty Acids as Oleic.	Acid Value.	Unsaponifiable Matter.	Mixed Fatty Acids.		
								Solidifying-point.	Melting-point.	Iodine Number.
Poppyseed oil		63.4	40°	13.1‡‡	160 to 277‖‖	0.7 to 11.0	0.43	16.2	20.5	139
Sunflower oil		72.2	25°			11.2	0.31		22 to 24	121 to 134
Corn oil	21.5	65.0	35°			1.35 to 2.86	1.7 to 20.6		18 to 20	119.5
Cottonseed oil	10.4	67.6 to 69.4	25°	7.6 to 18		0.0	0.73 to 1.64	32 to 35	35 to 38	111 to 115
Sesame oil	23 to 23.9	68	25°	11.5‡‡	0.47§§ to 5.75¶	0.23	0.95 to 1.32	22.9 to 23.8	26 to 32	110.45
Rape oil	17 to 25	68	25°	14.7	2.45¶	1.4 to 13.2	0.58 to 1.0	12 to 13	16 to 19	99 to 103
Black mustard oil		59.5	40°			1.36 to 7.35			16	109.6
White mustard oil		58.5	40°			5.4			15 to 16	95.3
Peanut oil		66 to 67.5	25°	3.4‡‡	1.45‖‖ to 8.28¶	1.2	0.54 to 0.94	29.2	27.7 to 32	96 to 103
Olive oil	15	62.4	25°	10.64	1.55 to 8.33‖‖	1.9 to 50	0.46 to 1.0	17.2 to 26.4	24 to 27	86 to 90
Lard oil		53.9	35°							
Cottonseed stearin								35.1	27 to 30	94
Cocoa butter		46 to 47.8	40°	2.8		1.1 to 1.88		48.3	48 to 50	33 to 39
Cocoanut oil		34	40°	0.9 to 12.3		5 to 50		22.5 to 25.2	25 to 27	8.4 to 9.3
Lard	9 to 11.8	48.6 to 51.2	40°	2.6	0.35 to 1.00¶¶	0.54 to 1.28	0.23	41 to 42	43 to 44	90 to 106
Beef tallow	6.1 to 7.2	49	40°	2.7 to 8.6		3.5 to 50		37.9 to 46.2	43 to 44	41.3
Mutton tallow	7.6 to 8.9	49	40°			1.7 to 14		40.15 to 48.02	49 to 50	34.8
Butter	6.6 to 9.5	41 to 42	40°	1.9 to 8.6		0.45 to 35.38			38 to 40	28 to 31

* Levallois.
† Gill and Hatch.
‡ Thompson and Ballantyne.
‖ Medicus and Scherer.
§ Girard.
¶ Hopkins.
** Gill.
†† Vary.
‡‡ Benedict and Ulzer.
‖‖ Crossley and Le Suer.
§§ Nördlinger.
¶¶ Wiley.

and palmitin will have crystallized out. This portion is then separated from the mother liquor by filtration through an alcohol-wet filter-paper, and the crystals finally treated as in the preceding section, being examined in a medium of olive or cottonseed oil.

OLIVE OIL.

Source.—Olive oil is derived from the fruit of the cultivated thornless olive tree, *Olea Europæa sativa*,* of which there are a great many varieties, originally grown in Asia Minor, Greece, Palestine, and southern Europe, and now cultivated extensively in California, Peru, and Mexico, as well as in Australia. Most of the olive oil of commerce, especially of the choicest varieties, is supplied by southern France, Spain, and Italy. The tree is an evergreen of slow growth and great longevity.

The ripe olive fruit is purple or purplish black in color; it is round or oval in shape, and from 2.5 to 4 cm. in diameter. The oil is contained in the parenchyma cells of the fruit suspended in a watery fluid. A thick skin incloses the fruit, and within is a kernel, which itself contains oil. The fruit contains from 40 to 60 per cent of oil. According to Brannt,† the average composition of the olive is as follows:

	Flesh, Per Cent.		Stone, Per Cent.		Seed, Per Cent.	
Oil	56.4		5.75		12.26	
Organic substances	16.70		85.89		79.38	
Nitrogen therein		1.10		2.50		2.16
Ash	2.68		4.16		2.16	
Water	24.22		4.20		6.20	
	100.00		100.00		100.00	

or no pressure. After the first pressing, the pomace is ground, treated with water, and again subjected to pressure. Several pressings in this manner may be carried out, each yielding an oil inferior to that preceding, the lowest grades being used for lubricants and in the manufacture of soap.

Nature and Composition.—The better grades of olive oil, suitable for table and medicinal purposes, possess a pleasant, bland taste, and a distinctive and agreeable odor, unmistakable in character for that of any other oil. The finest virgin oil is pale green in color, due to the presence of chlorophyll, which is closely associated with the oil globules in the cellular tissue of the fruit. Some varieties of olive oil are nearly colorless, while others are a deep golden yellow.

Olive oil contains 28% of solid glycerides, chiefly palmitin and a very small amount of arachin, and 72% of liquid glycerides, mainly olein with a little linolein. Stearin is practically absent.

Lewkowitch* states that olive oil differs from most vegetable oils in containing cholesterol but not phytosterol.

Gill and Tufts† show, as a result of numerous experiments, that olive oil is not thus exceptional, but that the unsaponifiable alcohol is phytosterol and not cholesterol.

Olive oil is very soluble in chloroform, benzol, and carbon bisulphide, but is sparingly soluble in alcohol. Five parts of ether will dissolve 3 parts of the oil.

ADULTERANTS.—As a rule the low grade olive oils are most subject to adulteration, by reason of the fact that it hardly pays to destroy or even modify the fine quality and delicacy possessed by a first class oil, which would inevitably be the result if even a small amount of foreign oil were added. Furthermore, if olive oil be slightly rancid or for any reason lacking in flavor, the admixture of a bland oil tends rather to minimize the fact.

The most common adulterant of olive oil in this country is naturally cottonseed oil, which is often substituted wholly for it. In Europe peanut oil is sometimes used both as an admixture and even as a substitute, since it possesses in itself a rather pleasant flavor, rendering it especially adapted for use as an adulterant. Other cheap oils used for this purpose are corn, mustard, poppyseed, rape, sesame, and sunflower

* Chem. Anal. of Oils, Fats, and Waxes, 2d ed., p. 452.
† Jour. Am. Chem. Soc., XXV, 1903, p. 498.

oil. The writer has also found in samples of alleged olive oil sold in Massachusetts cocoanut oil * and even fish oil.

Pure Olive Oil of the U. S. Pharmacopœia.—The requirements of the Pharmacopœia are as follows:

Specific gravity, 0.915 to 0.918 at 15° C. (59° F.).

When cooled to about 10° C. (50° F.), the oil should become somewhat cloudy from the separation of crystalline particles, and at 0° C. (32° F.) it should form a whitish, granular mass.

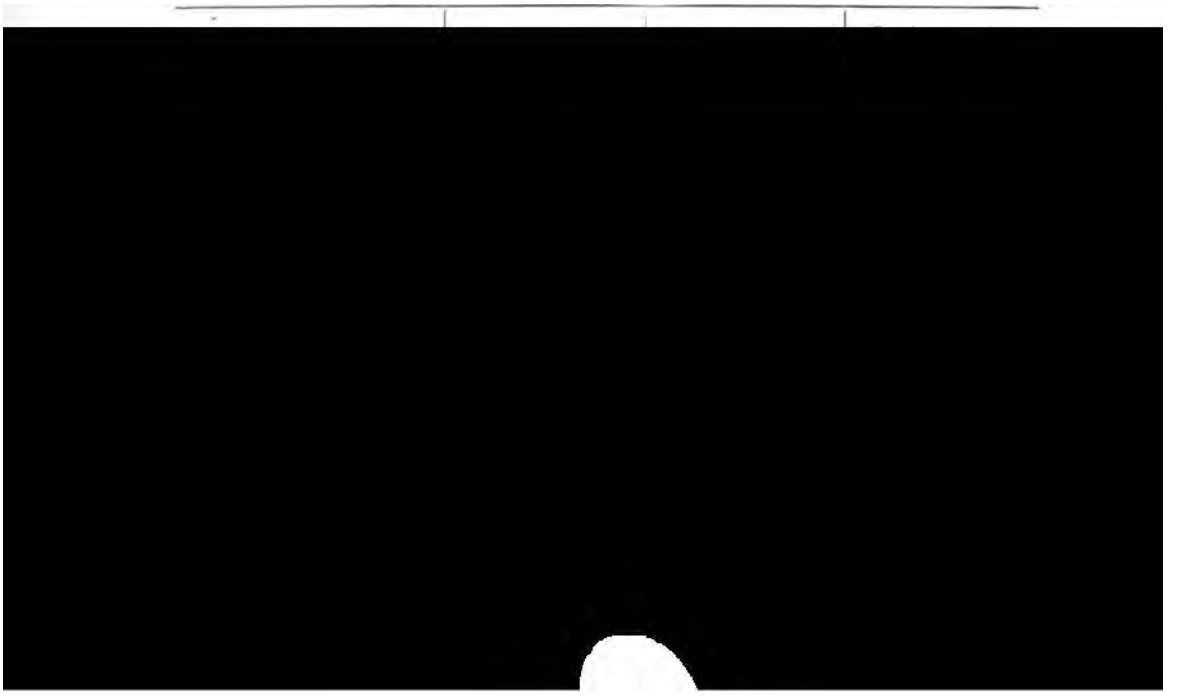
If 10 cc. of the oil be shaken frequently, during two hours, with a freshly prepared solution of 1 gram of mercury in 3 cc. of nitric acid, a perfectly solid mass of pale straw color should be obtained.

If 6 grams of the oil be thoroughly shaken in a test-tube for about two minutes with a mixture of 1.5 grams of nitric acid and 0.5 gram of water, then heated in a bath of boiling water for not more than fifteen minutes, the oil should retain a yellow color, not becoming orange or reddish brown, and, after standing at the ordinary temperature for about twelve hours, it should form a perfectly solid, light yellow mass (absence of appreciable quantities of cottonseed oil, and most other seed oils).

Olive oil should not show the cottonseed oil reaction with the Bechi test, p. 418, nor the sesame oil reaction with the Badouin test, p. 420.

Reaction with Strong Acid.—Pure olive oil, when shaken or stirred with an equal volume of concentrated nitric or sulphuric acid, turns from a pale to a dark-green color in a few minutes. If, under this treatment, a reddish to an orange, or brown coloration is produced, the presence of a foreign vegetable oil (usually a seed oil) is to be suspected.

Bach gives the following table showing the action of strong nitric acid on various oils:



The Zeiss Butyro-refractometer furnishes one of the most useful and easily applied preliminary means of judging the purity of the sample. If the reading is beyond the limits of pure olive oil, it at once indicates adulteration and often points to the particular adulterant. On the other hand, it is not always safe to assume the oil to be pure if the reading is correct, since mixtures of higher and lower refracting foreign oils may be so skillfully prepared as to read well within the limits of the pure oil on the refractometer scale. The refractometer reading of pure cottonseed oil is almost five degrees higher than that of pure olive.

READINGS ON ZEISS REFRACTOMETER OF OLIVE AND COTTONSEED OILS.*

Temperature (Centigrade).	Scale Reading.		Temperature (Centigrade).	Scale Reading.	
	Olive Oil.	Cottonseed Oil.		Olive Oil.	Cottonseed Oil.
35.0	57.0	61.8	25.5	62.4	67.5
34.5	57.2	62.1	25.0	63.0	67.9
34.0	57.4	62.3	24.5	63.3	68.2
33.5	57.7	62.5	24.0	63.6	68.5
33.0	58.0	62.8	23.5	63.9	68.8
32.5	58.3	63.0	23.0	64.2	69.1
32.0	58.5	63.2	22.5	64.5	69.4
31.5	59.0	63.6	22.0	64.8	69.7
31.0	59.2	64.0	21.5	65.1	70.0
30.5	59.4	64.2	21.0	65.4	70.3
30.0	59.9	64.5	20.5	65.7	70.6
29.5	60.1	64.9	20.0	66.0	70.9
29.0	60.3	65.1	19.5	66.3	71.2
28.5	60.6	65.3	19.0	66.6	71.5
28.0	60.9	65.7	18.5	66.9	71.8
27.5	61.1	66.0	18.0	67.2	72.1
27.0	61.5	66.5	17.5	67.5	72.4
26.5	62.0	67.0	17.0	67.8	72.7
26.0	62.2	67.3	16.5	68.1	73.0

The Elaïdin Test, in the case of pure olive oil, is very distinctive, since it yields by far the hardest elaïdin of all the common oils, and solidifies the most quickly.

Archbutt † shows the effect on this test of the mixture with olive oil of various proportions of rape and cottonseed oil, as follows:

Kind of Oil.	Minutes Required for Solidification at 25° C.	Consistency.
Olive oil	230	Hard but penetrable
" + 10% rape oil	320	Buttery
" + 20% "	From 9 to 11½ hours	"
" + 10% cottonseed oil.	" 9 " 11½ "	Very soft.
" + 20% "	More than 11½ "	" "

* Ann. Rep. Mass. State Bd. of Health, 1899, p. 647. † Jour. Soc. Chem. Ind., 1897, p. 447.

Cottonseed Oil as an adulterant is best detected by means of the Halphen or Bechi tests. Its presence in notable quantities increases the specific gravity, refractometer reading, and iodine number very materially. Its high Maumené figure is also distinctive.

Peanut Oil, when present to a considerable extent, betrays its presence by its peculiar bean-like flavor. Most of the constants of peanut oil lie within the limits of olive oil, with the exception of the refractometer reading, which is somewhat higher. A considerable admixture of peanut oil raises the refractometer reading perceptibly over that of pure olive. Its presence is best detected, however, by tests for arachidic acid (p. 424), taking care not to neglect the fact that pure olive oils of certain varieties have been known to respond to these tests.

Sesame Oil differs more particularly from olive in its higher specific gravity and iodine and Maumené numbers, and is readily detected by distinctive color tests (p. 420).

Rape Oil is characterized by a much lower saponification value and higher iodine number than olive.

Corn Oil differs materially from olive in its exceedingly high iodine number and refractometer reading. Its specific gravity and saponification numbers are also higher.

Lard Oil, when present in considerable quantity, is often rendered apparent by its characteristic odor on warming. Its low refractometer reading and iodine number are also distinctive.

Poppyseed Oil differs most widely from olive oil in its refractometric reading, its high dispersion, and its Maumené number, which in the case of poppyseed is 87° and of olive about 42°.

Cocoanut Oil in mixture with olive perceptibly raises the solidifying-

and then in succession try the various color reactions for sesame and rape oils. If all these are absent, and, by abnormal constants, or by color with nitric acid, there is reason to believe the oil is adulterated, determine carefully such of the constants as are most indicative, by their wide variation from olive, of poppyseed, mustard, and corn oils.

If all these oils are presumably absent, and either a high refractometer reading or a color reaction with nitric acid still indicates adulteration, peanut oil is more than likely to be present, and should be tested for either by Renard's or Bellier's method.

The edible oils and adulterants are arranged in order of their relative price about as follows:

Olive oil.
Lard oil.
Peanut oil.
Sesame oil.
Poppyseed oil.
Rape oil.
Corn oil.
Cottonseed oil.

COTTONSEED OIL.

Source and Preparation.—This oil, largely used as a table oil and as an adulterant of olive oil, is derived from seeds of the various species of the cotton plant, *Gossipium*, of which the most common are *G. herbaceum*, native to Asia, but cultivated extensively in southern Europe and in the United States, *G. religiosum*, in China and the East Indies, and *G. barbadense*, in the West Indies.

The seeds are in reality a by-product in cotton manufacture. In shape they are irregularly oval, measuring from 5 to 8 mm. greatest diameter. The seed skin or pod is covered with the fiber of the cotton.

The seeds are first cleaned and separated from dirt by sifting machines, and from the fiber by specially constructed gins, after which they are cut into small pieces, freed from their hulls, crushed between rollers, and afterward submitted to hydraulic pressure in bags to express the oil, which is clarified by filtration or refined. The refining consists in washing the crude oil with sodium hydroxide solution, whereby the impurities are dissolved and thus removed.

Nature and Composition of Seeds and Oil.—The seeds of the cotton plant are rich in oil, containing from 10 to 29 per cent, according to the

variety. Four samples of American cottonseed were found to be composed as follows, according to Brannt:*

Constituents.	South Carolina.	Georgia I.	Georgia II.	Georgia III.
Water	9.5	10.1	9.8	8.2
Cottonseed oil	20.1	16.2	17.1	19.6
Nitrogenous compounds	17.8	17.4	17.2	18.1
Ammonia-making compounds	2.3	2.9	3.2	3.7
Gum, sugar, and soluble starch	.8	.9	.7	.9
Cellulose, starch, and resin	26.2	27.4	26.1	20.7
Ligneous tissue	17.6	19.2	19.8	22.4
Ash (phosphate of lime, silica, alumina, iron, magnesia, potash, soda, etc.)	5.7	5.9	6.1	6.4
	100.0	100.0	100.0	100.0

Refined cottonseed oil is a pale-yellow oil of thick consistency, possessing a bland though pleasant taste and odor. It consists of the glycerides of oleic, stearic, palmitic, and linoleic acids, and evidently also a small content of hydroxyacids, though this has not been investigated as yet.

On cooling the oil to a temperature below 12° C. particles of solid fat will separate. At about 0° to −5° C. the oil solidifies. When the oil is brought in contact with concentrated sulphuric acid, a dark, reddish-brown color is instantly produced.

Cottonseed Stearin.—This product, used as an adulterant of lard as well as a substitute therefor, is obtained by submitting the oil to a low temperature, and subjecting the solid portion that separates out to pressure. It is a light-yellow fat, resembling butter in consistency.

Cottonseed oil from its cheapness is rarely adulterated by the admixture of foreign oil, but inferior grades are often sold for the best.

Bechi's Silver Nitrate Test.—*Hehner's Modification.*—Two grams of



sometimes show a slight reduction with Bechi's test. In cases of doubt it is well to apply the test on the fatty acids as follows:

*Milliau's Modification of Bechi's Test.**—Heat 20 grams of the sample with 30 cc. of alcoholic potash solution (20% potassium hydroxide in 70% alcohol), shaking at intervals till saponification is complete. Continue the heating for some minutes afterward until the alcohol is driven off, and dissolve the soap in 250 cc. of hot water. Add a slight excess of 10% sulphuric acid, and wash the separated fatty acids three times by decantation with water. Then proceed with a portion of the fatty acids as in Bechi's test.

Halphen's Test.—This is a much more delicate test for cottonseed oil than either of the preceding, as little as 2% of cottonseed oil being rendered apparent in olive oil. A mixture is made of equal volumes of amyl alcohol and carbon bisulphide in which 1% of sulphur has been dissolved. From 3 to 5 cc. of melted fat are mixed with an equal volume of the above reagent in a test-tube, loosely stoppered with cotton, and heated in a bath of boiling saturated brine for fifteen minutes. If cottonseed oil is present, a deep-red or orange color is produced. In its absence little or no color is developed.

Previous heating of the oil diminishes the delicacy of the Halphen test, and Holde and Pelgry † state that if cottonseed oil has been heated at 250° for ten minutes, it will fail to respond to the test. With care, however, a faint red color is even then obtainable.

SESAME OIL.

Sesame, otherwise known as benné, oil is pressed from the seeds of *Sesamum indicum* and *Sesamum orientale*, plants which are native to southern Asia, but now extensively cultivated in nearly all tropical countries. The larger portion of commercial sesame oil is manufactured in England, France, Germany, and Austria.

The seeds are yellow to dark brown, and in some cases black, inclined to the oval in form, the average longest diameter being about 4 mm.

The seeds are commonly subjected to cold pressure once, and afterwards twice pressed when warm, thus yielding three grades of oil. From 47 to 60 per cent of oil is contained in the seeds.

According to Brannt ‡ the composition of sesame seeds is as follows:

* Moniteur Scientifique, 1888, p. 366.
† Jour. Soc. Chem. Ind., 1899, 18, p. 711.
‡ Vegetable Fats and Oils, p. 251.

	Sesamum Orientale.		Sesamum Indicum.	
Oil.	55.63		50.84	
Organic substances.	30.95		35.25	
Protein therein		21.42		22.30
Nitrogen therein		3.39		3.56
Ash	7.52		6.85	
Water	3.90		7.06	
	100.00		100.00	

Sesame oil consists of the glycerides of oleic, stearic, palmitic, and myristic acids. It is golden yellow in color, free from odor, and possesses a delicate and characteristic flavor, on account of which the highest grades are by some considered equal to olive oil as a condiment. It is accordingly sold to some extent as an edible oil. It was formerly used as an adulterant of olive oil, but has of late years been largely displaced by cheaper oils for purposes of adulteration. When cooled to -3° C., sesame oil congeals to a yellowish-white mass. Concentrated sulphuric acid converts it into a brownish-red jelly.

Adulterants to be looked for in sesame oil are cottonseed, poppy-seed, corn, and rape oils.

Tocher's Test.*—One gram of pyrogallic acid is dissolved in 15 cc. of concentrated hydrochloric acid and mixed with 15 cc. of the sample in a separatory funnel. After standing for a minute, the aqueous solution is withdrawn and boiled. If sesame oil is present, the solution shows a red coloration by transmitted, and blue by reflected, light.

Badouin's Test.†—Dissolve 0.1 gram of cane sugar in 10 cc. of hydrochloric acid (specific gravity 1.20) in a test-tube, and shake thoroughly with 20 grams of the oil to be tested for one minute. Then allow the



the agency of the levulose produced by the inversion of the sugar by hydrochloric acid. As furfurol is the chief product of the reaction between levulose and hydrochloric acid, it was substituted as follows: Dissolve 2 grams of furfurol in 100 cc. of 95% alcohol, and shake 0.1 cc. of this solution in a test-tube with 10 cc. of the oil to be tested and 10 cc. of hydrochloric acid (specific gravity 1.20) for half a minute. The aqueous layer, on settling out, will be colored deep red, if sesame is present.

Or 0.1 cc. of the alcohol furfurol solution is mixed with 10 cc. of oil and 1 cc. of hydrochloric acid in a separatory funnel, shaken well, and the separation aided by the addition of chloroform, which causes the aqueous layer, showing color with sesame oil, to float.

Since furfurol produces with hydrochloric acid alone a violet coloration, it is necessary to use it in dilute solution as above.

RAPE OIL.

Rape or colza oil is expressed from the seeds of the Brassica or rape-plant, of which there are three principal varities, *Brassica napus*, *B. compestris*, and *B. rapa*, one or another of which are cultivated in nearly every country of Europe, excepting Greece. Large amounts are also grown in India and China. The seeds are small, round grains, from 2 to 2.5 mm. in diameter, yielding from 30 to 45 per cent of oil. The seeds, according to Brannt,* have the following average composition:

	Fresh Seeds.		Old Seeds.	
Oil	36.80		38.50	
Organic substances	49.30		53.25	
Nitrogen therein		2.50		3.11
Ash	4.80		3.90	
Water	9.10		4.35	
	100.00		100.00	

In the process of preparation the seeds are first crushed, and the oil removed by pressing or extraction. The crude oil is of a brownish-yellow color, and when fresh is almost free from taste and smell, so that it serves, when cold pressed, as an edible oil, or an adulterant of such oils. It develops a disagreeable and peculiar taste and odor on long standing, due to the presence of certain albuminous and mucilaginous substances which it contains. These may be removed by refining, usually by treatment with sulphuric acid, but the refined oil has an unpleasant taste and odor.

* Vegetable Fats and Oils, p. 240.

The principal components of rape oil are the glycerides of stearic, oleic, erucic, and rapic acids. The chief adulterants are cottonseed and poppyseed oils.

Palas Test for Rapeseed Oil.*—Mix in the cold 30 cc. of a 1% solution of fuchsin, 20 cc. of sodium bisulphite (specific gravity 1.31), 200 cc. of water, and 5 cc. sulphuric acid. If the sample of oil to be tested be shaken with the reagent, a rose-red coloration is obtained in the presence of rape oil, said to be delicate to the extent of detecting 2% of the oil in mixtures.

CORN OR MAIZE OIL.

Corn oil is derived from the seed of the American grain *Zea mays*, or Indian corn, the constitution of the yellow and white varieties of which is, according to Andés,† as follows:

	Yellow Corn, Per Cent.		White Corn, Per Cent.	
Organic matter	82.93		80.76	
Starch		61.95		62.23
Albuminoids		10.71		9.62
Ash	1.32		1.04	
Water	9.50		10.60	
Oil.	6.25		7.60	
	100.00		100.00	

Nearly all the oil is contained in the germ of the seed, the oil constituting in fact over 20% of the germ. Corn oil consists chiefly of the glycerides of palmitic and oleic acids. There is some doubt as to the presence of stearin. It is golden yellow in color, and possesses a pleasant odor and taste, resembling in flavor freshly ground grain.

unlike most vegetable oils, contains cholesterol. Olive oil was long supposed to be unique as a vegetable oil in containing this substance. Hopkins, on the assumption that cholesterol occurs in corn oil, suggested that a test for corn oil as an adulterant of certain vegetable oils lay in the identification of cholesterol.

Gill and Tufts * claim that, while the alcohol of corn oil is not phytosterol, neither is it cholesterol, but a third substance, known as sitosterol,† occurring in wheat and rye.

There are no color reactions identifying corn oil as such. Its presence in other oils is indicated only by its influence on the various constants, the iodine number and refractometric reading especially being much higher than those of other edible oils.

PEANUT OIL.

Peanut or arachis oil is obtained from the seeds of the *Arachis hypogæa* (peanut, ground nut, or earth nut) cultivated in most tropical countries, notably in South America, China, India, and Japan. The plant is a creeping herb, developing its blossoms in the axes of the leaves. The fruit buds grow down into the earth, where the fruit is ripened, forming the well-known peanuts of commerce, the composition of which, according to Brannt, is as follows:

	Per Cent.		Per Cent.	
Oil	37.48		to 41.63	
Organic substances	52.86		" 53.12	
Albumin therein		27.25	"	27.85
Ash	2.43		" 2.50	
Water	7.37		" 2.75	
	100.00		100.00	

Peanut oil is composed chiefly of the glycerides of oleic, palmitic, hypogæic, and arachidic acids. The oil is extracted by pressure, the first cold-drawn oil being practically colorless, and possessing a pleasant taste suggestive of kidney beans. It is especially adapted for use as a salad or table oil. A second pressure of the moistened residue from the first yields an inferior oil, yellowish in color, also somewhat used for edible purposes, and sometimes commercially called "butterine oil."

* Jour. Am. Chem. Soc., XXV, 1903.

† Burian, Monatsh. Chem., 18, 1897, p. 551.

Adulterants of peanut oil are cottonseed, poppyseed, rape, and sesame oils. Very little pure peanut oil is found in commerce in the United States. It is to be looked for as an adulterant of French and Italian olive oils.

Characteristic Tests.—Peanut oil, when pure or nearly pure, may as a rule be readily identified from other oils. When present in large admixture in other oils it is not difficult to detect, but when only a small amount is present, in olive oil for instance, its detection becomes a more troublesome matter.

This difficulty arises from the fact that the constants of peanut oil are nearly the same as those of olive, with the single exception of the refractometric reading. Furthermore, there is no readily applied color test identifying peanut oil.

All the other common adulterants of olive oil, as cottonseed, sesame, corn, poppyseed, and rape oils, are readily identified, when present in small amounts, either by special color tests, or by reason of the fact that certain of their constants differ very widely from those of olive oil. Much more care and precaution are necessary in dealing with small admixtures of peanut oil than with almost any other adulterant.

The Renard Test* has long been in use for detecting and estimating peanut oil in mixtures, but cannot be regarded as entirely satisfactory, since it has been found to give the reaction for arachidic acid in cases of certain varieties of pure olive oil, as well as of cottonseed and sesame oils, known to be free from peanut oil. Under these conditions it can hardly be regarded as infallible, and by many is deemed useful chiefly as a confirmatory negative test in proving the absence of peanut oil from a doubtful sample.

Tolman† claims to be able to obtain positive results by carrying out



the soap when cold adheres to the sides of the flask. The water and excess of lead acetate can then be poured out, leaving the soap in the flask. Wash by shaking and decantation, first with cold water and then with 90% alcohol. Add 200 cc. of ether, cork the flask, and allow to stand with occasional shaking till the soap is disintegrated, after which boil on a water-bath under a reflux condenser for five minutes. Cool the soap solution down to a temperature between 15° and 17°, and allow it to stand for about twelve hours.

Filter and wash the precipitate with ether, after which the soap in the filter is washed back into the original flask with a stream of hot water acidulated with hydrochloric acid.

Add an excess of hydrochloric acid and enough hot water to make a volume of from 300 to 400 cc., and cool in ice water till the fatty acids have hardened and separated from the lead chloride. Then filter, wash the precipitate with water, and, after draining dry, pour slowly through the filter 25 cc. of boiling 95% alcohol. The crystals of arachidic acid separate out as the solution cools. To further purify, filter and wash the precipitate into a flask with a stream of alcohol, measuring the volume used, which should be about 20 cc. Cork the flask, shake, and again filter, and wash with 70% alcohol. Finally dissolve off the precipitate with boiling absolute alcohol, evaporate to dryness in a tared dish, dry and weigh. To the weight add 0.0025 gram for each 10 cc. of 95% alcohol used in the crystallization and washing, if done at 15° C., and 0.0045 gram for each 10 cc. if done at 20°.

The approximate amount of peanut oil is found by multiplying the weight of arachidic acid by 20.

Arachidic acid crystals thus obtained should be examined microscopically, and the melting-point determined. For arachidic acid obtained in this manner the melting-point should lie between 71° and 72° C.

Methods of J. Bellier.*—*Qualitative Test.*—Saponify 1 gram of the oil with 5 cc. of an alcoholic potash solution containing 85 grams potassium hydroxide per liter of strong alcohol, conducting the saponification in a small Erlenmeyer flask on the water-bath. After saponification, boil for two minutes, neutralize with dilute acetic acid, using phenolphthalein as an indicator, and cool by setting the flask in water at a temperature of from 17° to 19°. After a short time, a precipitate nearly always comes down. Then add to the solution 50 cc. of 70% alcohol, containing 1% by volume of strong hydrochloric acid (specific

* Ann. Chim. Anal., 1899, 4, p. 49; Zeits. für untersuch. Nahr., 1899, 2, p. 726.

gravity 1.20). Cork the flask, shake vigorously, and again cool by setting the flask in the above cooling-bath. In the absence of a precipitate, the oil may be pronounced free from peanut. If 10% or more of peanut oil is present, a more or less characteristic precipitate forms, and often with less than 10% a cloudiness in the solution is perceptible after standing between 17° and 19° for half an hour. Pure olive oil remains perfectly clear as a rule.

A few varieties of olive oil from Tunis especially high in solid fat acids, as well as cottonseed oil and sesame oil, give similar turbidity on the addition of the 70% alcohol. To distinguish between these oils and peanut oil, heat the mixture on the water-bath till complete solution takes place, and again cool to 17° to 19°. In the case of peanut oil the cloudiness or precipitate again occurs to the same extent as before, while in the other cases the solution should remain clear or nearly so.

Quantitative Determination.—Saponify 5 grams of the oil with 25 cc. of the above alcoholic potash solution in a 250-cc. Erlenmeyer flask, neutralize exactly with acetic acid, and cool quickly in water. After standing an hour, pour upon a 9-cc. filter and wash the precipitate with 70% alcohol containing 18% by volume of hydrochloric acid, the temperature of the solution being not less than 16° nor more than 20°. Continue the washing till the wash water no longer shows turbidity when diluted with water.

Dissolve the precipitate in 25 to 30 cc. of hot 95% alcohol, dilute with water until the alcohol is 70%, let stand in water at 20°, filter, wash with 70% alcohol, dry at 100°, and weigh.

Bellier states that he has recognized with certainty as small an admixture as 2% of peanut oil by this method.



Mustard oil is an alleged adulterant of edible oils, though by no means a common one.

POPPYSEED OIL.

This oil is obtained from the seeds of the opium poppy (*Papaver somniferum*), native in the countries east of the Mediterranean, and cultivated extensively for opium and for oil in all parts of Europe, Asiatic Turkey, Persia, Egypt, India, and China. Most of the oil of commerce comes from France and Germany.

There are two chief varieties of poppy, the black (*P. nigrum*) and the white (*P. album*), the finest oil being produced from the white. The seeds are somewhat flattened in form and kidney-shaped, yielding from 40 to 60 per cent of oil. According to Brannt the seeds have the following composition:

	White Poppy-seed.		Black Poppy-seed.	
Oil	55.62		51.36	
Organic substances	32.11		35.14	
Protein therein		16.89		17.50
Ash	3.42		4.00	
Water	8.85		9.50	
	100.00		100.00	

The oil is obtained by crushing the seeds and applying pressure. The best grade of cold-drawn oil is pale yellow in color, possessing a pleasant taste when fresh, and being practically free from odor. Lower grades shade into deeper yellow and even reddish color, possessing a strong taste and odor. Poppyseed oil is much used in Europe as a table oil, and does not readily turn rancid. It is composed of the glycerides of stearic, palmitic, and linoleic acids. Poppyseed oil has been used to some extent as an adulterant of olive oil. It is itself not infrequently adulterated with sesame oil.

SUNFLOWER OIL.

Sunflower oil is derived from the seed kernels of the plant of the same name (*Helianthus annuus*), originally grown in Mexico, but now cultivated most extensively on a commercial scale in southern Russia.

According to S. M. Babcock * the composition of sunflower seeds is as follows:

	Air-dry.	Dried.
Water	12.68	
Ash	3.00	3.43
Albuminoids (N×6.25)	15.88	18.19
Crude fiber	29.21	33.45
Nitrogen-free extract	18.71	21.43
Fat (ether extract)	20.52	23.50
	100.00	100.00

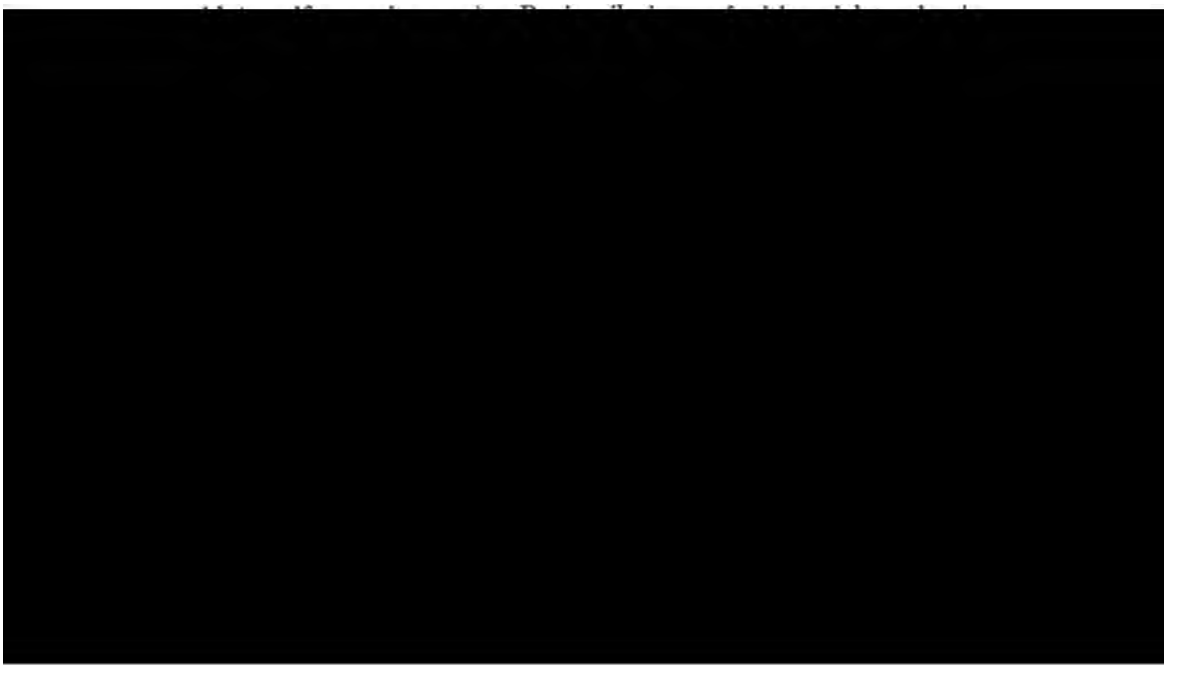
The seeds are long, black, and oval in shape, yielding from 18 to 28 per cent of oil. The liquid fatty acids of sunflower oil consist for the most part of linoleic, but little oleic acid being found.

The seeds are first shelled, then crushed, and finally submitted to pressure both cold and hot.

Sunflower oil is pale yellow in color, has a mild, pleasant taste, and is nearly free from odor. The cold-drawn oil is the variety most used for edible and culinary purposes in Russia, and as an adulterant of olive oil. Its use as an adulterant is, however, limited, and the writer has no knowledge of its having been found in olive oils used in the United States.

ROSIN OIL.

Rosin oil is prepared by the distillation of common rosin, and is an alleged adulterant of olive oil. It may be detected when present by shaking 1 to 2 cc. of the sample with acetic anhydride while warming. Cool, remove the anhydride by a pipette, and add a drop of sulphuric



oil polarizes from +30 to +40 on the cane sugar scale, while other oils have a reading between +1 and −1.

COCOANUT OIL.

Cocoanut oil is the fat expressed from the kernels of the cocoanut or fruit of the cocoa palm (*Cocos nucifera*), indigenous to the South Sea Islands and to the East-Indian archipelago, but grown in many tropical countries.

It is sometimes known as "copra oil," from the copra or pulp, which contains from 60 to 70 per cent. of fat. According to Brannt, the composition of the pulp is as follows:

	Indian Copra.		African Copra.	
Oil	68.75		66.80	
Organic substances	23.65		25.25	
Albuminous substances		9.16		10.20
Ash	1.45		1.50	
Water	6.15		6.45	
	100.00		100.00	

In the preparation of the oil the moist copra is separated from the shell, crushed in mortars and subjected to pressure, yielding a milky mass. This is then heated in boilers, and the oil which rises to the surface is removed by skimming.

In some localities the pulp is first dried and then pressed.

Cocoanut oil is usually white and possesses a mild taste and pleasant odor. The cold-drawn Malabar oil is of greenish color, and is used by the natives as an edible oil or substitute for butter. This variety is seldom found in commerce.

Cocoanut oil contains, besides palmitin and olein, large proportions of myristin and laurin. Unlike the other vegetable oils, it contains also notable quantities of the glycerides of the volatile fatty acids caproic, capric, and caprylic, hence the high saponification value and Reichert number are the two most distinguishing constants. The oil is rarely adulterated.

Cocoanut oil easily becomes rancid. According to Andés, crystals of cocoanut oil appear under the microscope as a thick network of long needles.

COCOA (CACAO) BUTTER.

This preparation is not, properly speaking, in itself an edible fat. It is a by-product in the manufacture of cocoa, being removed by pressure from the crushed and ground cocoa nibs. The fat in cocoa beans varies from 36 to 50 per cent. The expressed fat is yellowish white, of a tallow-like consistency, has a pleasant taste and an odor suggestive of chocolate. It keeps a long time without turning rancid. In composition it consists of the glycerides of stearic, palmitic, and lauric acids, with traces of the glycerides of arachidic and butyric acids.

Its demand for pharmaceutical purposes is, however, sufficiently great to render the use of cocoa-butter as an adulterant of food-fats extremely rare. It should be borne in mind as a possible adulterant in examining various oils.

It is subject to adulteration with paraffin, tallow, and cottonseed stearin.

TALLOW.

The rendered fats of various animals, especially the cow and sheep, constitute what is generally known as tallow. The untreated fatty tissues are more properly known as suet, the tallow being the clear fat separated entirely by heat from the cellular material.

Tallow consists almost entirely of olein, palmitin, and stearin. Mutton tallow is usually, but not always, harder than beef tallow.

Excepting in the manufacture of material for oleomargarine, wherein the heart and caul fats of beef are almost exclusively used, the fats from different parts of the animal are not, as a rule, separated.



Butter fat is of extremely complex composition, containing a larger variety of glycerides than any other fat. Besides olein, palmitin, and stearin, the usual glycerides of the insoluble or fixed fatty acids found in most fats, butter contains notable quantities of the glycerides of a number of the volatile fatty acids, chief among which are butyrin, caproin, caprin, and caprylin, to which are due its distinctive taste, and which by exposure to light and air readily become decomposed into their fatty acids—butyric, caproic, capric, and caprylic, respectively. This decomposition in butter causes, or, more properly speaking, accompanies, what is commonly known as "rancidity."

The process of separation of butter fat into its component glycerides is a matter of extreme difficulty, and results obtained by different chemists vary widely. Separation has been attempted by fractional distillation, by methods depending on the difference in chemical affinity of the various acids, and on the difference in solubility of the various lower homologues in water at different temperatures.*

According to Browne, the composition of butter fat is as follows:

Acid.	Percentage of Acid.	Percentage of Triglycerides.
Dioxystearic	1.00	1.04
Oleic	32.50	33.95
Stearic	1.83	1.91
Palmitic	38.61	40.51
Myristic	9.89	10.44
Lauric	2.57	2.73
Capric	0.32	0.34
Caprylic	0.49	0.53
Caproic	2.09	2.32
Butyric	5.45	6.23
Totals	94.75	100.00

Upwards of 300 analyses of butter are summarized by König in the following table:

	Water, Per Cent.	Fat, Per Cent.	Casein, Per Cent.	Milk, Per Cent.	Sugar, Per Cent.	Lactic Acid, Per Cent.	Salts, Per Cent.
Minimum	4.15	69.96	0.19		0.45		0.02
Maximum	35.15	86.15	4.78		1.16		15.08
Mean	13.59	84.39	0.74	0.50		0.12	0.66

* Browne, A Contribution to the Chemistry of Butter Fat, Jour. Am. Chem. Soc., 21, 1899, p. 807.

ANALYSES OF BUTTER.

Preparation of the Sample.—(*A. O. A. C. Method.*) *—If large quantities of butter are to be sampled, a butter-trier or sampler may be used. The portions thus drawn, about 500 grams, are to be perfectly melted in a closed vessel at as low a temperature as possible, and when melted, the whole is to be shaken violently for some minutes till the mass is homogeneous, and sufficiently solidified to prevent the separation of the water and fat. A portion is then poured into the vessel from which it is to be weighed for analysis, and should nearly or quite fill it. This sample should be kept in a cold place till analyzed.

Water.—About 2 grams of the sample are weighed in a flat-bottomed platinum dish such as is used for determining water in milk, and the dish and its contents kept in contact with the live stream of a water-bath till a constant weight is attained.

Fat.—This may be determined either directly or indirectly. For the direct determination, a weighed amount of the sample, from 2 to 3 grams, is first dried at 100° in sand or asbestos, contained in a thin and fragile round-bottomed evaporating-shell (Hoffmeister's Schälchen). If desired, the moisture may be determined in this connection by loss in weight after drying. The shell is afterwards inclosed in a piece of fat-free filter-paper, and crushed in pieces between the fingers in such a manner as to avoid loss. The pieces are gathered in a mass and folded together in the filter-paper to form a packet of a size readily transferable to a Soxhlet extractor, in which the fat is removed in the usual manner and weighed, after drying, in a tared flask.

Or the fat may be indirectly determined as follows: The residue



Ash.—About 10 grams of the butter are transferred to a tared flask of about 100 cc. capacity, and its exact weight determined. About 50 cc. of ether or naphtha are then added, and the fat dissolved by shaking, pouring the solution upon an ash-free filter or on a tared Gooch. Wash out the flask and the residue on the filter with fresh portions of ether or naphtha, dry the residue, and if filter-paper is used, transfer to a porcelain dish and burn to a white ash in a muffle or over a free flame, or burn the Gooch directly. Cool in a desiccator and weigh.

Casein.—Proceed with 10 grams of the fat as in the preceding section up to the point of washing the residue upon a filter. Dry the residue on the filter and determine the nitrogen according to the Gunning method, making a blank test on the filter and reagents:

$$\text{Nitrogen} \times 6.37 = \text{casein.}$$

Milk Sugar and Lactic Acid compose most of the undetermined matter remaining after deducting from the total solids the sum of the fat, casein, and ash. Determine milk sugar, if desired, in an aqueous extract of the butter by Fehling's solution.

Determination of Salt.—In a tared dish or beaker weigh out about 5 grams of butter, taking a gram or so at a time from different parts of the sample. Add hot water to the weighed part, and after it has melted, the contents of the dish are poured into a separatory funnel, shaken and allowed to stand till the fat collects at the top, after which the underlying aqueous solution is drawn off into an Erlenmeyer flask, leaving the fat in the funnel bulb. Hot water is again added, and from ten to fifteen extractions are made, using about 20 cc. of water each time, all the water being collected in the Erlenmeyer flask.

A few drops of a solution of potassium chromate are then added for an indicator, and the sodium chloride volumetrically determined by a standard silver nitrate solution.

Salted butter contains from 0.5 to 6% of salt.

Examination of Butter Fat.—The butter fat is best obtained free from curd and salt by filtering when hot, the sample being best melted in a beaker on the water-bath. The water, with the curd and salt, will settle to the bottom. The clear fat is then filtered at a temperature not exceeding 50° C., and subjected to such examination as may be desired to determine its purity.

U. S. Standard Butter Fat has a Reichert-Meissl number not less than 24 and a specific gravity not less than 0.905 at $\frac{40^\circ}{40^\circ}$ C.

ADULTERATION OF BUTTER.

The artificial coloring of butter is an art practiced for so many years, and is so far in accord with the popular demand, that it can hardly be considered as an adulteration. The most recent custom of adding preservatives other than salt to butter is, however, very properly considered in most localities as reprehensible, unless the character and amount of the preservative be made clear to the purchaser by a suitable label.

The most common and time-honored sophistication is the substitution in whole or in part of foreign fat, as in the case of oleomargarine, and, more recently, in the fraudulent sale of renovated or process butter for the freshly made article.

U. S. Standard Butter is butter containing not less than 82.5% of butter fat.

ARTIFICIAL COLORING MATTER IN BUTTER.—Formerly carrot juice and annatto were used almost entirely as butter colors. The carrot furnished to the farmer a ready means of coloring his dairy butter, and its use was long in vogue for this purpose, before the commercial butter colors were available. Other vegetable colors, such as turmeric, marigold, saffron, and safflower, are said to have been used for this purpose, but, with the possible exception of turmeric, the writer is not aware of authentic cases in which they have been found in recent years. While annatto as a butter color is still in use, it is rapidly giving place to various oil-soluble, azo coal-tar colors, which are admirably adapted to the purpose. All butter colors are now put on the market in solution in oil, usually cottonseed in this country and sesame in Europe.

Detection.—*Martin** devised a general scheme, applicable for the detection of various colors in butter. His reagent consists of a mixture of a



Turmeric is to be suspected, if ammonia turns the alcoholic extract brown; *marigold*, if silver nitrate turns it black, and *annatto*, if on evaporating the alcoholic solution to dryness and applying to the residue a drop of concentrated sulphuric acid, a greenish-blue coloration is produced.

Turmeric is further tested for in the residue from the alcoholic extract as above obtained, by boiling the residue in a few cubic centimeters of a dilute solution of boric acid (or a solution of borax acidulated with hydrochloric acid), and soaking a strip of filter-paper therein. On drying the paper, if it assumes a cherry-red color, turning dark olive by dilute alkali, the presence of turmeric is assured.

Carrotin (the coloring matter of the carrot root) does not impart its color to the alcohol layer in Martin's test. Moore * has pointed out this exception, and shown that while with carrotin present the alcohol layer in Martin's test remains colorless, as in the case of uncolored butter, that when, however, a drop of very dilute ferric chloride is added, and the test-tube shaken, if carrotin be present, the alcohol will gradually absorb the yellow color from the butter. Care must be taken to avoid an excess of ferric chloride, as very little of this reagent will suffice.

Allen states that a butter color commercially known as "carrotin" consists in reality of 1 part of annatto in 4 parts of oil.

Detection of Annatto in Butter.—Treat 2 or 3 grams of the melted and filtered fat (freed from salt and water) with warm, dilute sodium hydroxide. After stirring, pour the mixture while warm upon a wet filter, using to advantage a hot funnel. If annatto is present, the filter will absorb the color, so that, when the fat is washed off by a gentle stream of water, the paper will be dyed straw color. It is well to pass the warm alkaline filtrate two or three times through the fat on the filter to insure removal of the color.

If, after drying the filter, the color turns pink on application of a drop of stannous chloride solution, annatto is assured.

Detection of Coal-tar Colors in Butter.—*Geisler's Method.*†—A few drops of the clarified fat are spread out on a porcelain surface and a pinch of fullers' earth added. In the presence of various azo-colors, a pink to violet-red coloration will be produced in a few minutes. Some varieties of fullers' earth react much more readily with the azo-dyes than do others. In fact some do not respond at all. When once a satisfactory sample of this reagent is obtained, a large stock should be secured of the same variety.

Low's Method.‡—A small amount of material to be tested is melted

* Analyst, 11, p. 163. † Jour. Am. Chem. Soc., 20, 1898, p. 110. ‡ *Ibid.*, 20, p. 889.

in a test-tube, an equal volume of a mixture of 1 part of concentrated sulphuric acid and 4 parts of glacial acetic acid are added, and the tube is heated nearly to the boiling-point, the contents being thoroughly mixed by shaking; the tubes are set aside, and after the acid solution has settled out it will have been colored wine-red in the presence of azo-color, while with pure butter fat, comparatively no color will be produced.

Doolittle's Method for Azo-colors and Annatto.*—The melted sample is first filtered. Two test-tubes are taken and into each are poured about 2 grams of the filtered fat, which is dissolved in ether. Into one test-tube are poured 1 or 2 cc. of dilute hydrochloric acid, and into the other about the same volume of dilute potassium hydroxide solution. Both tubes are well shaken and allowed to stand. In the presence of azo-dye, the test-tube to which the acid has been added will show a pink to wine-red coloration, while the potash solution in the other tube will show no color. If annatto has been used, on the other hand, the potash solution will be colored yellow, while no color will be apparent in the acid solution.

PRESERVATIVES AND THEIR DETECTION.

Fresh or unsalted butter and renovated butter are often found with an added preservative, the one most commonly used for this purpose being the so-called "boric mixture" (borax and boric acid) already discussed under milk adulteration. Salted butter is occasionally though not so often found preserved. Other preservatives used in butter are formaldehyde and salicylic and sulphurous acids. These latter are, however, rarely found.

Boric Acid.—This, if present, is best detected in the aqueous solution

faint, the aqueous solution is acidified slightly with hydrochloric acid and the turmeric-paper applied as before. If borax be present to an appreciable extent, the red color will now be quite marked, even though not appearing before. In other words, testing with turmeric-paper without acidifying with hydrochloric acid shows, according to Richmond, a slight coloration due to the free acid alone, while the more intense color formed by first acidifying is due to the combined acid or borax.

Determination of Boric Acid.—Ten grams of the butter fat are weighed in a beaker and transferred with hot water to a separatory funnel in which the fat is extracted with 10 to 15 portions of hot water as described on page 433. The combined aqueous extract is evaporated to dryness in a platinum dish, the residue made alkaline, and ignited at a dull red heat. Boil the ash with water, filter, and wash with hot water, keeping the volume of the filtrate under 60 cc. Make sure that the solution is perfectly neutral to methyl orange by treatment, if necessary, with sulphuric acid and tenth-normal alkali, add 30 cc. of glycerin, a few drops of the phenolphthalein indicator, make up to 100 cc., and titrate with tenth-normal sodium hydroxide according to Thompson's method (p. 669).

Butter being practically free from phosphates, the preliminary treatment for removing phosphoric acid in Thompson's method may be omitted.

Formaldehyde.—The aqueous solution from which the fat of the butter melted at low temperature has been poured off, is added to some milk previously found free from formaldehyde, and the test for the latter with hydrochloric acid and ferric chloride is tried directly in the milk.

Salicylic Acid.—*Detection.*—See method No. 2 for detection in milk, page 143.

Determination of Salicylic Acid.—Method of the Paris Municipal Laboratory.—Repeatedly exhaust 20 grams of butter in a separatory funnel with a solution of sodium bicarbonate, thus obtaining soluble sodium salicylate, if salicylic acid be present. Acidulate the aqueous extract with dilute sulphuric acid, and extract with ether. Evaporate the ether, and to the residue add a little mercuric nitrate, forming a precipitate nearly insoluble in water. Filter this off, wash the precipitate with water, and decompose into free salicylic acid with dilute sulphuric acid. Redissolve in ether, evaporate the solvent as before, and dry the residue at a temperature of 80° to 100°. Extract the residue with petroleum ether, dilute the ethereal liquid with an equal volume of 95% alcohol,

and titrate with tenth-normal alkali, using phenolphthalein as an indicator.

1 cc. of tenth-normal alkali = 0.0138 gram salicylic acid.

Sulphurous Acid.—The aqueous liquid, separated from the butter fat, is distilled, and the distillate treated with bromine water and barium chloride. A precipitate on the addition of the latter reagent indicates the presence of sulphurous acid or a sulphite in the butter.

Renovated or Process Butter.—This product is also variously termed "boiled," "aerated," and "sterilized" butter. There are various modifications of the process of manufacture, but the object is to melt up and treat rancid butter in such a manner that for a time at least it is sweet. The following manner of treatment is typical, and shows in the main the necessary steps in carrying out the process, though details of manipulation vary in different localities.

The butter is melted in large tanks surrounded with hot water jackets at a temperature varying from 40° to 45° C. By this means the curd and brine settle to the bottom, whence they are drawn off, while the lighter particles rise to the top in the form of a froth or scum and are removed by skimming.

The clear butter fat is then, as a rule, removed to other jacketed tanks, and, while still in a molten condition, air is blown through it, which removes the disagreeable odors. The melted fat is then churned with an admixture of milk (more often skimmed) till a perfect emulsion is formed, after which it is rapidly chilled by running into ice cold water, with the result that it becomes granular in form. It is then drained and "ripened" for some hours, after which it is worked free from excess of milk and water, salted, and packed.

Under some state laws this product, to be legally sold, must conform



composed entirely of fat other than that derived from milk or cream, they are comparatively rare. As a rule the oleomargarine of commerce is composed of refined oleo oil, usually churned up with neutral lard, milk, and a small amount of pure butter, the whole being salted and sometimes colored to resemble butter.

Oleo oil is prepared from the fat of beef cattle somewhat as follows:* Immediately after the animals are killed the fresh intestinal and caul fat are removed and placed in tanks of water at a temperature of about 80° F. From this water they are transferred to other tanks of cold water and chilled until all animal heat is removed. The fat is then cut or hashed into small pieces and melted at about 150° F. in jacketed steam kettles, until the clear oil is entirely separated from the connective tissue.

This oil is then drawn off into vats, which, on account of the appearance of the oil on cooling, are called graining or seeding vats, where it is allowed to stand for twenty-four hours or more at a temperature of about 85° F. From these vats the semi-solid emulsion of oil and stearin is dipped into cloths, which are folded and placed in a press between sheets of metal and subjected to powerful pressure. By this means the oil is separated from the stearin, and is drawn into casks for export or for manufacture into oleomargarine. Large quantities are annually exported to Holland, where oleomargarine is manufactured, and either sold for consumption in that country, or re-exported to other countries in Europe.

The oleo oil thus expressed is a mixture of olein and palmitin. When first prepared, it is a clear amber-colored fluid, free from odor or fatty taste. It is packed in tierces, and, when opened at ordinary temperature, is a light-yellow solid.

The further process of manufacture of oleomargarine consists in the main of mixing the oleo oil as above obtained with varying proportions of neutral lard, milk, and genuine butter, with or without added coloring matter, and churning the mixture at a temperature above the melting-point of the fats, the neutral lard having previously been cured for at least forty-eight hours in salt brine. Occasionally small quantities of other vegetable oils, as cottonseed, peanut, or sesame, are included in the above mixture. After the churning, the whole mass is cooled by contact with ice water. The chilled mass is drained, and afterwards salted, worked, and given much the same treatment as butter.

* Report on Oleomargarine, Its Manufacture and Sale, 19th An. Report, Mass. St. Bd. of Health, 1887.

The composition of commercial oleomargarine varies between the following limits:

Oleo oil	20 to 25%
Neutral lard	40 " 45%
Butter	10 " 25%
Milk, cream, salt, etc.	5 " 30%

The artificial coloring matters employed for oleomargarine are the same as in the case of butter, and are similarly tested for.

In many states oleomargarine cannot be legally sold when colored to resemble butter. Under other state laws coloring matter is allowable. The federal law and most state laws prescribe the most rigid rules for marking packages containing oleomargarine, with a view to affording the utmost protection to the producer of butter against the fraudulent substitution therefor.

Healthfulness of Oleomargarine.—Under the directions of the Massachusetts Board of Health,* a large number of artificial digestion experiments were made to show the relative nutritive value of butter and oleomargarine, and at the same time the wholesomeness of oleomargarine as a food was carefully investigated. The general conclusions reached were that, when comparing the best grades of both products, there is little if any difference between butter and oleomargarine on grounds of digestibility, while a good oleomargarine is much to be preferred to a poor butter from a nutritive standpoint. As to its wholesomeness, a large number of experts consulted were unanimous in expressing their favorable opinions of oleomargarine as a healthful article of food.

When sold on its own basis in accordance with the law, it forms an excellent cheap substitute for butter. It is only when fraudulently sold



of limited value, except in so far as it indicates that the fat has first been melted and afterwards solidified.

From the fact that oleo oil and neutral lard form by far the larger portion of the mixture known as oleomargarine, the glycerides that make up the fat of the latter are chiefly those of the insoluble fatty acids, stearic, oleic, and palmitic. The percentage of volatile fatty acids present in oleomargarine is very small, and the presence of these volatile acids is due entirely to the admixture of butter which it contains. This furnishes the most ready means of distinguishing chemically between the two products, and, as indicated by the Reichert number, is the chief reliance of the analyst for court evidence.

Incidentally, as will be seen by the accompanying table, the refractometer reading, the iodine number, the saponification equivalent, and the specific gravity are all useful constants in indicating points of difference between the two fats, it being understood that in oleomargarine, as in butter, the fat for examination is melted and separated by filtration or otherwise from the curd, salt, and other constituents.

CONSTANTS OF BUTTER FAT AND OLEOMARGARINE.

	Specific Gravity at 100°.	Water.	Insoluble Acids.	Soluble Acids.	NaCl.	Albuminoids.	Curd.	Koettstorfer's Equivalent.	Reichert Number.*	Butyro-refractometer Reading.	Temperature.
Butter fat:											
Maximum.	0.870§	31.55‡	89.60†	5.94‡	5.62‡	.875‡	3.10‡	233§	15.8†‖	47.7†	35°
Minimum	.867§	4.44‡	85.63†	3.00‡	0.00‡	.175‡	0.40‡	222§	12.4†	44.8†	35°
Oleomargarine.											
Maximum.	.8625§	11.69‡	95.45†	1.16‡	3.64‡	.350‡	0.74‡	204§	5.5†	54.8†	35°
Minimum	.8585§	9.34‡	92.46†	0.12‡	2.30‡	.3063‡	0.63‡	192§	0.5†	53.0†	35°

* Number of cubic centimeters N/10 alkali neutralizing volatile acids in 2.5 grams fat.
† From analyses made in Mass. State Board of Health laboratory.
‡ From analyses made in laboratory of U. S. Dept. of Agric., Bur. of Chem.
§ From analyses by A. H. Allen.

The constants for varying mixtures of butter with foreign fat as found by Villiers and Collin ‖ are tabulated on page 442.

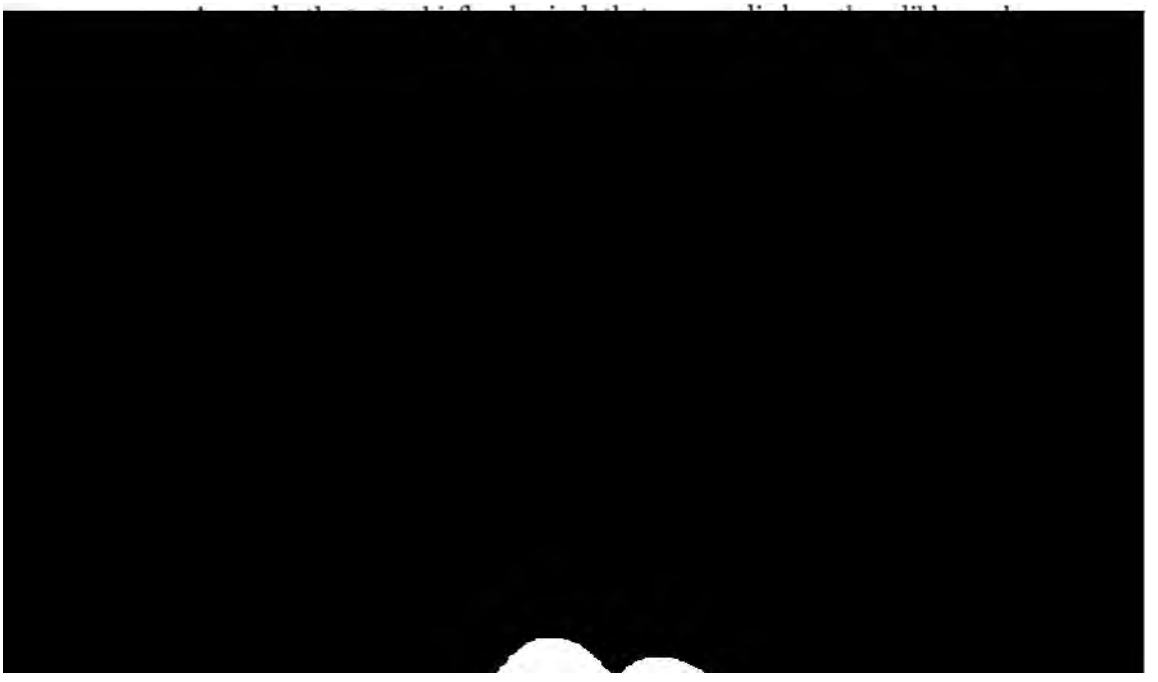
Odor and Taste. — It is easy with a little practice to become so accustomed to the odor and taste of oleomargarine, as to be able to pass judgment with considerable confidence by these senses alone, whether a sample in question is oleomargarine or butter. The distinction is rendered more apparent by melting a portion of the sample on the water-bath. If the product is butter, either fresh or renovated, the butyric odor of the melted fat is very characteristic, while the melted oleomargarine

‖ Les Substances Alimentaires, p. 731.

not only is lacking in the butyric odor (a negative property), but possesses a distinctive "meaty" smell peculiar to itself, which, while not unpleasant, is unmistakable. The flavor of oleomargarine to one experienced in distinguishing between the two products is very apparent. This flavor, slight though it is, might be compared to that of cooked meat.

	Hehner's Number.	Soluble Acids.	Koettstorfer's Equivalent.	Volatile Acids.
Pure butter.	88	5	224	26
Butter, 95%; foreign fat, 5%	88.35	4.8	222.6	24.7
" 90% " " 10%	88.70	4.5	221.2	23.4
" 85% " " 15%	89.05	4.3	219.8	22.2
" 80% " " 20%	89.40	4.1	218.4	20.9
" 75% " " 25%	89.75	3.9	217	19.6
" 70% " " 30%	90.10	3.6	215.6	18.3
" 65% " " 35%	90.45	3.4	214.8	17.1
" 60% " " 40%	90.80	3.2	212.8	15.8
" 55% " " 45%	91.15	3	211.4	14.5
" 50% " " 50%	91.50	2.7	210	13.2
" 45% " " 55%	91.85	2.5	208.6	12
" 40% " " 60%	92.20	2.3	207.2	10.7
" 35% " " 65%	92.55	2.1	205.8	9.4
" 30% " " 70%	92.90	1.8	204.4	8.1
" 25% " " 75%	93.25	1.6	203	6.9
" 20% " " 80%	93.60	1.4	201.6	5.6
" 15% " " 85%	93.95	1.2	200.2	4.3
" 10% " " 90%	94.30	0.9	198.8	3
" 5% " " 95%	94.65	0.7	197.4	1.8
Foreign fat.	95	0.5	196	0.5

Distinguishing between Butter, Process Butter, and Oleomargarine.— With the increased occurrence in the market of the commercial product known as "process" butter, especially in localities where its sale is restricted or regulated by law, it becomes incumbent on the analyst to distinguish it from the other products which it resembles.



which are applied to the separated fat, freed from other substances, will serve to distinguish between oleomargarine and butter, whether "process" butter or otherwise, since the "processing" or "renovating" of butter does not change the character of its fat sufficiently to materially alter these constants.

It is best, therefore, for purposes of routine preliminary separation to submit *all* samples to the "foam" test and to examine them by the butyro-refractometer.* These tests alone, which are very quickly and readily applied, will rarely fail to separate into the three classes, butter, process butter, and oleomargarine, the products under examination, after which such confirmatory tests as are desired are made on adulterated samples.

The Butyro-refractometer.—This instrument, as its name implies was primarily intended by Zeiss for the examination of butter, and, while its use has been extended for work with other fats and oils, its construction is such as to show particularly a distinction between butter and oleomargarine by the appearance of the critical line of the fat. This mode of differentiation is due to the peculiar construction of the double prism, which shows differences of dispersive power by different appearances of the critical line. The prisms are so constructed that the critical line of pure butter is colorless, while margarine and artificial butter, which have greater dispersive powers than natural butter, show a blue-colored critical line. But anomalies in the color, both with pure butter and mixtures, are more or less observable, which render it impossible to draw a sharp line between adulterated and genuine butter. The appearance of a blue fringe may, however, be a useful factor in cases of suspected adulteration.

The following particulars respecting the application of the refractometer for analysis of butter are contained in a paper of Dr. R. Wollny of Kiel,† who assisted in the construction of the instrument. The readings of the refractive indices of a large number of butter samples taken at 25° C. by Dr. Wollny have been directly reduced to scale divisions and yield the following equivalents:

* Out of the large number of samples of butter and oleomargarine examined on the butyro-refractometer in the author's laboratory during eight years, he has never found a single instance where the instrument failed to show the difference between the two products.

† Dr. R. Wollny, Schlussbericht über die Butteruntersuchungsfrage, Milchwirthschaftlicher Verein, Korrespondenzblatt, No. 39, 1891, p. 15.

Older papers on butter tests by refraction of light will be found in: Mueller, Rep. d. analyt. Chemie, 1886, pp. 346, 366. Skalweit, Milchzeitung, 1886, 15, p. 462. Wollny, Ueber die Kunstbutterfrage, Leipzig, 1887, p. 50.

Natural butter...(1.4590–1.4620):49.5–54.0 scale divisions
Oleomargarine...(1.4650–1.4700):58.6–66.4 " "
Mixtures (artificial
butter) .....(1.4620–1.4690):54.0–64.8 " "

Limit of Scale Reading for Pure Butter.—Whenever in the refractometric examination of butter at a temperature of 25° C. higher values than 54.0 are found for the critical line, these samples will, according to Wollny, by chemical analysis always be found to be adulterated; but with all samples in which the value for the position of the critical line does not reach 54.0 chemical analysis may be dispensed with, and the samples may be pronounced to be pure butter. Wollny suggests, as a means of removing all chances of adulterated butter escaping detection, that the above limit be placed still lower, and that all samples exhibiting values exceeding 52.5 (at a temperature of 25° C.) be set aside for chemical analysis.

In calculating the position of the critical line for other temperatures than 25° C. allow per 1° C. variation of temperature a mean value of 0.55 scale division.* The following table, which has been compiled in this manner, shows the values corresponding to various temperatures, each value being the upper limit of scale divisions admissible in pure butter:

Temperature.	Scale Division.	Temperature.	Scale Division.	Temperature.	Scale Division.	Temperature.	Scale Division.
45°	41.5	40°	44.2	35°	47.0	30°	49.8
44°	42.0	39°	44.8	34°	47.5	29°	50.3
43°	42.6	38°	45.3	33°	48.1	28°	50.8
42°	43.1	37°	45.9	32°	48.6	27°	51.4
41°	43.7	36°	46.4	31°	49.2	26°	51.9



line may be determined at the moment when the butter begins to set. In this case he gives fifty-four as the highest admissible number for the critical line of pure butter.

No sharp distinction is apparent between pure and renovated butter on the refractometer.

Special Thermometer for the Butyro-refractometer.—Instead of employing the ordinary thermometer, as shown in Fig. 91, a special thermometer (Fig. 98) has been devised for work both with butter and with lard. This instrument has two scales, arranged side by side, one for butter and one for lard, each of which indicates at once the highest allowable reading for the pure fat, corresponding to the temperature at which the observation is made, which, however, need not be noted.

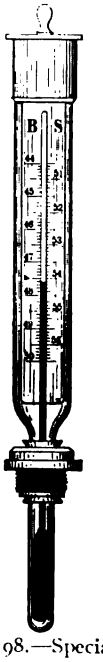


FIG. 98.—Special Butyro-refractometer Thermometer for Butter and Lard.

If the scale reading of the instrument, as observed through the telescope, differs materially from the reading of the special thermometer, the fat under examination is undoubtedly adulterated, or, in the case of butter, a higher reading indicates oleomargarine. The special thermometer thus indicates the highest permissible number for pure butter.

The Reichert or Reichert-Meissl Number * is by far the most important single determination in establishing proof of the character of the sample, whether butter or oleomargarine, for evidence in court, and in such cases this determination is indispensable. The result is conclusive, excepting in those rare instances where the admixture of foreign fat is so small as to cause the Reichert number to approximate that of pure butter. In common instances of creamery butter and commercial oleomargarine the Reichert number shows a very marked distinction (see table, p. 441).

It is difficult to fix a minimum figure below which, in doubtful cases, a sample may be pronounced impure by reason of admixture with foreign fat. In general, however, a Reichert number under 10 would be almost sure to show adulteration, though instances are on record where butter of known purity has shown a Reichert number even lower than this. It is in fact rare that pure butter has a Reichert number under 12.

* The writer prefers to carry out this process on 2.5 grams of the butter fat, expressing thus the *Reichert number*, this being practically half the Reichert-Meissl number, which is based on the use of 5 grams.

Stebbins * gives the maximum, minimum, and average of the Reichert number obtained by him on 317 samples of unadulterated butter, some of which were of low grade, as follows: Maximum, 18.2; Minimum, 11.2; Average, 14.7.

As a rule little difference is apparent between pure and "renovated" samples as regards their Reichert number.

Vieth has shown that the Reichert number of butter is generally a trifle lower after it becomes rancid.

Specific Gravity.—Skalweit has shown that the specific gravity of butter and oleomargarine relative to each other varies with the temperature at which it is taken, the difference between the two growing less and less as the temperature increases above 35°. The greatest variation being at 35°, he recommends this temperature as the best at which to make the determination.

The Foam Test, also known as the "boiling" or "spoon" test.† This, though originally intended as a household test, is in reality one of the very best laboratory methods of separating pure butter samples from renovated butter and oleomargarine. A small lump of the sample (from 3 to 5 grams) is heated in a large spoon over a Bunsen flame, turned very low, stirring constantly during the heating. Genuine butter, under these conditions, will boil quietly, but with the production of considerable froth or foam, which will often swell up over the sides of the spoon, when, just after boiling, the latter is raised from the flame. Renovated butter or oleomargarine, under this treatment, will bump and sputter noisily like hot grease containing water, but will not foam.‡ Another point of difference is that on removing the spoon from the flame and observing the character of the curdy particles, in the case of genuine butter these particles of curd will be very small and finely divided in



condition and cooled therein, whereas oleomargarine, being foreign to milk fat, will, under like conditions, refuse to diffuse itself naturally in milk as a medium.

About 50 cc. of well-mixed sweet milk are heated nearly to boiling in a beaker, and from 5 to 10 grams of the fat sample are added. The mixture is then stirred, preferably with a small wooden stick, until the fat is melted. The beaker is then placed in a dish of ice cold water, and the stirring continued till the fat reaches the solidifying-point, at which period, if the sample is oleomargarine, the fat can readily be collected by the stirrer into one lump or clot, but, if butter, it cannot be so collected, but remains in a granulated condition, distributed through the milk in small particles. It is not necessary to keep up the stirring through the entire term of cooling, but to begin stirring before the fat starts to solidify, which should require from ten to fifteen minutes after the mixture is placed in cold water.

This test, if carefully carried out, shows a marked distinction between butter, whether pure or renovated, and oleomargarine. Under certain conditions, as when the cooling is too rapid, samples of renovated butter fat will sometimes show a slight tendency to clot together as in the case of oleomargarine, but to no such extent as the latter.

The author's experience with this test has shown it to be very reliable not only in identifying oleomargarine from butter, but in nearly every case renovated butter can be distinguished from genuine. As a rule, genuine butter fat, even after cooling to the solidifying-point, shows the greatest tendency to emulsionize with the milk when stirred, without adhering to the wooden rod, and is slow to come to the surface when the stirring is stopped. Renovated butter fat, when stirred in the cold milk, almost instantly gathers in a film on the surface of the milk when the stirring is stopped, without emulsionizing. It does not clot together like oleomargarine, but it tends to adhere to the wooden rod.

Patrick * recommends the use of skimmed or partially skimmed milk, and heats to the boiling-point after the fat has been introduced into the hot milk.

Examination of the Curd.—The curd of genuine butter is made up largely of such of the milk proteids as are insoluble in water and hence pass into the cream when separated. These proteids form a gelatinous mass in the butter, readily clotting together when the fat is melted. On the other hand, the curd of process butter, which is, as it were, artificially

* Farmer's Bulletin, No. 131.

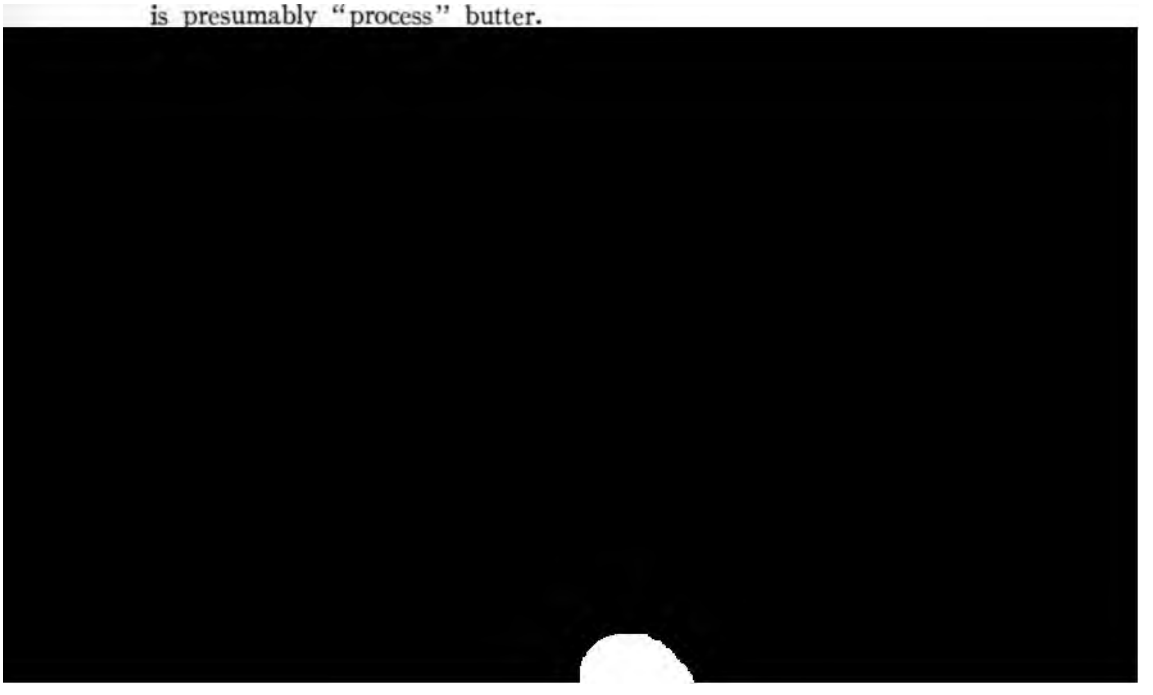
derived from the entire or skim milk used in its manufacture (in order to replace the natural curd which has been removed in the "purifying" process), differs from the proteids of cream in that it is granular and flaky, consisting chiefly of coagulated casein. Hence the distinction noted as to the appearance of the curd in the foam test.

For the same reason, if beakers containing pure and renovated butter are melted on the water-bath, the curd of the pure sample will settle at once, or in a very few minutes, to the bottom after melting, leaving a comparatively clear supernatant fat. The renovated sample will nearly always fail to settle out clear, even after standing on the water-bath for half an hour or more, but will still be cloudy throughout the mass, due to particles of non-cohesive, floating curd.

In the case of oleomargarine, the curd of which is composed partly of pure butter curd (from cream proteids) and partly of the proteids of the milk with which it is churned, the cloudiness of the fat on melting depends on the relative proportion of milk proteids, and in general is not especially characteristic.

Identification of the Source of the Curd.*—Half fill a small beaker with the sample and melt on the water-bath. Decant as much as possible of the fat and pour the rest, consisting largely of the water, salt, and curd, upon a wet filter. Acidify the filtrate, which contains the salt and soluble proteids, with acetic acid and boil. If the sample is pure butter, only a slight milkiness is found, indicating absence of albumins, whereas, in the case of process butter, a white, flocculent albuminous precipitate is produced.

Apply to the filtrate also Liebermann's test for albumin; i.e., add strong hydrochloric acid. If a violet coloration is produced, the sample is presumably "process" butter.



that of the renovated, wherein often large and opaque patches of curd are frequently distributed throughout the field.

When a renovated butter sample, mounted as above, is viewed *by reflected light*, for which purpose the microscope mirror is turned so as not to transmit light through the instrument, one sees a very dark and scarcely perceptible field; but the opaque patches of curd above referred to are strikingly apparent as white masses against a dark background.

With Polarized Light.—It has already been stated that the microscope is useful in showing whether or not a fat has been melted, the crystalline structure of the fat once melted and afterward cooled being rendered apparent, especially when viewed by polarized light. This fact has long been known and put to practical use in the identification microscopically of butter and oleomargarine.*

When viewed by polarized light between crossed Nicols under a low magnification, pure butter not previously melted should show no crystalline structure, being uniformly bright throughout, and, if the selenite plate be used, should present an evenly colored field, entirely devoid of fat crystals. On the other hand, with process butter or oleomargarine, both of which have been melted and subsequently cooled, the crystalline structure should be marked, showing with polarized light a more or less mottled appearance, and a play of colors with the selenite.

Various conditions enter in to affect the reliability of the polarized light test. It is nearly always possible in cold weather to observe these distinctions in practice, as above described, in a sharp and striking manner. Figs. 269, 270, and 271, Pl. XXXVIII, show typical fields of the three products with crossed Nicols and selenite plate. The appearance of pure butter is perfectly blank, while oleomargarine presents a much more mottled appearance than renovated butter. Such well-defined points of variation as are shown in Plate XXXVIII are not always to be seen in practice, even in the hands of an expert. Pure butter sometimes exhibits a somewhat mottled field, due to a slight crystallization at some period in its history. In the summer-time, for instance, when butter melts so easily at ordinary temperature, these distinctions between pure and adulterated samples as shown by polarized light are by no means as satisfactory as in the winter.

Great care should be taken on this account, on the part of the collector of samples as well as the analyst, to keep the sample from melting under ordinary conditions before it is examined.

* Hummel, Jour. Am. Chem. Soc., 22, p. 327; Crampton, loc. cit., supra, p. 703.

Hess and Doolittle's Method of Examining the Curd.*—A convenient portion of the sample of suspected butter is melted in a beaker, as much of the fat as possible is decanted off, and the remaining curd, washed free from fat with ether, is poured out on a glass plate and dried. A sample of pure butter is treated in like manner by way of comparison. When examined under a very low magnification of from 3 to 6 diameters, the curd from the pure sample will be seen to be non-granular and amorphous in appearance, while, in the case of renovated butter, the curd will appear very coarse grained and mottled.

Zega's Test for Oleomargarine.†—A portion of the filtered fat is poured into a test-tube and kept for two minutes in a boiling water-bath. 1 cc. of this fat is then measured with a hot pipette into a 50-cc. tube containing 20 cc. of a mixture of 6 parts ether, 4 parts alcohol, and 1 part glacial acetic acid. The tube is stoppered, shaken well, and cooled in water at 15° to 18° C. In the case of pure butter fat, the solution remains clear for some time, a slight deposit being apparent only after standing an hour or more. With oleomargarine, a deposit is evident in a very short time, and in ten minutes a heavy precipitate comes down. With 10% of oleomargarine in butter, a separation occurs in about fifteen minutes. When a few solid particles have separated out, they are withdrawn and examined under the microscope. With genuine butter, long narrow rods appear, sometimes pointed at the ends, often bent, and grouped as a rule centrally in star-shaped bundles. Oleomargarine presents an appearance of bundles of fine needles, closely packed to form masses frequently resembling sheaves and dumb bells in shape.

Identification of Various Oils and Fats. — Cottonseed oil may be recognized, if present in butter or its substitutes, by the Halphen test, and sesame oil by the Badouin test. Peanut oil is tested for by the

vent decomposition. In one sample made for export to Guadeloupe he found over 10% of glucose.

For its detection or estimation 10 grams of the sample are weighed out and transferred to a separatory funnel with hot water, and shaken out with successive portions of hot water. These are combined, and the aqueous extract made up to 250 cc. The reducing sugar may be determined by Fehling's solution or by polarization, using in the latter case alumina cream as a clarifier. While a slight reduction should be disregarded, any considerable reduction may be undoubtedly ascribed to glucose.

Adulterants of Oleomargarine.—This product is liable to adulteration not only by the use of inferior and unwholesome fat, but by the admixture in some cases of paraffin.* This sophistication is made manifest, if an appreciable amount of the adulterant has been used, by the high melting-point and the low saponification number, as well as by the low specific gravity. If a clear saponification is impossible under ordinary conditions, paraffin is to be suspected. It may be separated and quantitatively determined as described on p. 409.

LARD.

Nature and Composition.—Lard is the fat of hogs, separated by heat from the scraps or containing tissues. The choicest or highest grade of lard is known as leaf lard, and is derived from the fat which surrounds the kidneys. A comparatively small part of the lard of commerce is, however, strictly speaking, pure leaf lard. Most of it is derived from the whole fat of the animal by rendering, by the aid of steam under pressure, either in open kettle or in closed tanks, the former being used more often for rendering lard on a small scale, and the latter being the most common commercial method.

Next to the leaf, the fat from the hog's back is considered the best in quality, after which is graded, in the order named, the fat from the head, the region of the heart, and the small intestines, the last two grades constituting what is commonly known as "trimmings."

Good lard is white and granular, having the consistency of salve. It has an agreeable, characteristic odor and taste.

The leaf or kidney fat furnishes also the source of the so-called neutral lard, already mentioned as an ingredient of oleomargarine. The leaf,

* Geissler, Jour. Am. Chem. Soc., 21, p. 605.

being first chilled and finely ground, is placed in the kettle and rendered at a temperature of from 40° to 50° C., at which heat only a portion of the lard separates. This portion is, while melted, washed with water containing salt or dilute acid, and forms the neutral lard, a product almost entirely free from odor. The remainder of the leaf is then transferred to the closed tank and subjected for some hours to steam under pressure at a temperature of 230° to 290° F., the resulting lard being graded as pure leaf lard.

The composition of the mixed fatty acids of lard is thus calculated by Twitchell:

Linoleic acid..... 10.06%
Oleic acid..... 49.39%
Solid acids (by difference), stearic and palmitic .. 48.55%

Lewkowitsch * gives the following constants for American lards made from fat from different parts of the animal:

Fat from	Specific Gravity at 100° C. (Water at 15° C. = 1.)	Iodine Value.	Maumené Number at 40° C.	Melting-point, Bensemann's† Method.		Refractive Index.
				Temp. C. of Incipient Fusion.	Melted to a Clear Drop.	Butyro-refractometer at 40° C.
Head	0.8637	66.2	33	24	44.8	52.6
	0.8629	66.6	32	24	44.8	52.5
	0.8631	65.0	34	24	45.0	52.0
Back	0.8611	61.5	37	28.5	48.5	52.4
	0.8621	65.0	35	28.5	48.5	51.8
	0.8616	65.1	38	31.5	46	51.9
Leaf	0.8637	62.2	..	26	45	51.4
	0.8615	59.0	..	29	44	50.2
	0.8700	63.0	30	28.5	44.5	52.0
Foot	0.8589	68.8	..	24	40	44.8

gravity, due to varying conditions of pressure and temperature. It has a pleasant, though somewhat bland taste, and is used to some extent as an edible oil. It is used in France as an adulterant of olive oil, and with the Maumené, elaïdin, and nitric acid tests, it behaves much like olive oil.

According to the U. S. Pharmacopœia, the specific gravity of lard oil should be from 0.910 to 0.920 at 15° C.

At a temperature a little below 10° C. it should form a semi-solid white mass.

When it is brought in contact with concentrated sulphuric acid, a dark reddish-brown color should instantly be produced.

Lard oil should not respond to the Bechi test for cottonseed oil.

If 5 cc. of the oil, contained in a small flask, be mixed with a solution of 2 grams of potassium hydroxide in 2 cc. of water, then 5 cc. of alcohol added, and the mixture heated for about five minutes on a water-bath with occasional agitation, a perfectly clear and complete solution should be formed, which, on dilution with water to the volume of 50 cc., should form a transparent, light-yellow liquid, without the separation of an oily layer (absence of appreciable quantities of paraffin oils).

Adulterants of lard oil are cottonseed and corn oils.

Compound Lard.—The article so extensively made and sold under this name is a mixture consisting usually of lard stearin, beef stearin, and cottonseed oil. Sometimes no lard whatever is present, but only a mixture of beef and cottonseed stearins.

Lard stearin is the residue left in the cloths after the lard oil has been removed by pressure (p. 452).

Beef stearin is, similarly, the residue from which oleo oil has been expressed (p. 439). The cottonseed oil used is highly refined, and finally decolorized by mixing with fullers' earth and filtering.

U. S. Standards.—*Standard Lard and Standard Leaf Lard* are lard and leaf lard respectively, free from rancidity, containing not more than 1% of substances other than fatty acids, not fat, necessarily incorporated therewith in the process of rendering, and standard leaf lard has an iodine number not greater than 60.

Adulteration of Lard.—The mixture known as "compound lard" is quite commonly fraudulently sold for pure lard. Indeed, the adulterants of lard usually met with are cottonseed oil or stearin and beef stearin. Other oils said to have been used as adulterants are peanut, sesame, corn, and cocoanut. Formerly water was incorporated into

the fat to such an extent as to materially cheapen it, but this sophistication is now rare. Moisture is determined as in the case of butter.

The Butyro-refractometer Reading.—The refracting degree of cottonseed oil on the butyro-refractometer is about 8.9 in excess of the standard refraction of lard, while that of beef tallow is about 3.8 less than the standard. If, therefore, the refractometer reading is unusually low, the presence of beef stearin is to be suspected; if unusually high, cottonseed oil should be looked for. A mixture of the two adulterants with pure lard such as is found in "compound lard," may sometimes, though not often, be found to give refractometric readings within the limits of pure lard.

Detection of Foreign Oils.—Cottonseed oil is best detected by the Halphen test. A *very slight* color reaction should not be taken as proof positive of the admixture of cottonseed oil, since it has been found that the fat of hogs fed on cottonseed meal gives a slight reaction with both the Bechi and the Halphen tests. Sesame and peanut oils are detected by their special tests. Corn oil is indicated by the abnormally high refractometric reading and iodine number, cocoanut oil by the high Reichert number and the high saponification equivalent.

Beef stearin is difficult to identify chemically, but is usually distinguished by a microscopical examination of the fat after crystallization as follows:

The Microscopical Examination of Lard.—From 2 to 5 grams of the fat are dissolved in 10 to 20 cc. of ether * in a test-tube, and the solution allowed to stand 12 hours or over night at about 20° C., the test-tube being loosely stoppered with cotton. The crystals obtained vary considerably with the condition of heat, amount of solvent, rate of crystallization, etc., so that the operator had best vary these conditions till



to have them slightly oleaginous, so that when applied to the slide no mountant need be used. In this case the crystals seem to stand out in wider contrast to the background than when cottonseed oil, the usual medium, is used.

If the crystals are, however, in a pulverulent condition, a drop of alcohol can be used as a mountant, or oil, as preferred. Mounted under a cover-glass they are examined under various powers of the microscope.

Figs. 272 and 273, Pl. XXXIX, show the typical appearance of pure lard stearin from a leaf lard of known purity, and Figs. 276, 277, and 278 illustrate beef stearin. These figures show distinctive crystallization of each form under the best conditions. The lard stearin crystals when thus obtained are flat rhomboidal plates cut off obliquely at one end, and are grouped irregularly, as if thrown carelessly together. The beef stearin crystals, on the other hand, are cylindrical rods or needles, often curved, with sharp ends, and are arranged as shown in fan-shaped clusters. Conditions of crystallization are frequently such as not to show the sharp distinctions noted above. Both forms of crystals are at times apt to gather in clusters that at first sight appear somewhat similar, and are often misleading as to their true character. It is found almost invariably that the beef stearin crystals gather in clusters, radiating from a common center or point, often with a peculiar twisted appearance, breaking up into little fans. Lard crystals, it is true, do not always lie flat in irregular groups as shown in Fig. 272, but, as in Fig. 274, form clusters that, unless studied carefully, might at first sight be considered as identical with the fan shapes of the beef stearin already described. It will be seen, however, that if the best possible conditions are attained, the crystals of lard, instead of radiating from a point, are arranged more like feathers or alternate leaves on a branch, each crystal being given forth from another close at hand. Moreover, the lard crystals are themselves straight and not curved, the apparent curve in the appearance of the clusters being, on careful examination, especially under high power, seen to be chiefly due to several of these straight crystals arranged at angles to each other.

Even when the highest powers of the microscope are applied to the beef stearin crystals, they will always appear as cylindrical, sharp-pointed rods, some straight, others curved; while with the lard crystals they should be capable of showing the thin, flat, oblique-ended structure when examined with higher powers, even when they are arranged in the feathery clusters, the apparently pointed ends of some of the crystals

being due to the fact that the plates are viewed edgewise. This is apparent in Fig. 275, in which the crystals are magnified to 480 diameters.

According to Belfield, who was one of the earliest to employ the microscope for identification of foreign fat in lard, it is possible to detect well-defined crystals of both lard and beef stearin in mixtures crystallized out in the above manner from ether. Later investigators, however, find difficulty in getting both kinds of crystals in the final deposit, it being the more common experience that the character of the final crystals from a mixture of the two fats more often tends to one or the other forms of crystallization. Repeated crystallizations may change the character of the crystals and a number of such crytallizations should therefore be made before final judgment is passed.

The Iodine Number (p. 380).—This test is generally prefigured by the refractometer. Cottonseed oil will absorb about 108% of its weight of iodine, while beef fat will absorb about 37%.

ANALYSES OF SAMPLES ILLUSTRATING TYPES OF LARD, LARD SUBSTITUTES, AND MIXTURES.

	Nitric Acid Test.	Crystallization.	Bechi Reaction.	Butyro-refractometer.			Iodine Number.	Conclusion.
				Temperature, Degrees.	Reading.	Variation from Standard.		
A	Slight color.....	Lard stearin	None	42.5	49.7	+0.1	58.1	Lard
B	Red	" "	"	42	50	+0.2	59.9	"
C	Slight color	" "	"	41.5	50.1	+0.0	58.7	"
D	" "	" "	"	43	50	+0.6	63.7	"
E	" "	" "	"	41.3	51	+0.8	64.6	"
F	" "	" "	"	42	50.5	+0.7	64.8	"
G	" "	" "	"					Lard

refractometer number, while the presence of tallow will result in an abnormally low refraction. But both adulterants may be present and a normal refraction result. In such a case the positive detection of one of them, such as the cottonseed oil by the Bechi or Halphen test, will indirectly show the presence of the other (tallow), and this indirect proof will be confirmed by crystallization.

Samples A, B, and C give reactions corresponding to normal, pure lard. D, E, and F show somewhat high refractometer and iodine numbers, but give no direct reaction for cottonseed oil by the Bechi test. G, although showing low iodine and refractometer numbers, gives no evidence of the presence of tallow by crystallization. In fact, the crystals from this sample proved under all circumstances to be most clearly typical of pure lard, broad and flat plates with obliquely cut ends.

This sample was, in fact, pure leaf lard. It is generally true that a stiff, strictly pure leaf lard, which both by its consistency and by its low iodine and refractometer numbers might suggest the presence of beef fat, shows on crystallization much more definitely characteristic lard stearin than does a whole-hog lard, whose iodine and refractometer numbers are more nearly the normal standard.

In distinction from such leaf lard, a sample which may have a similar consistency and iodine and refractometer numbers, but which is composed of a whole-hog lard of a comparatively high iodine number, together with beef fat, gives unmistakable proof of its adulteration by its crystallization.

REFERENCES ON EDIBLE OILS AND FATS.

ANDÉS, L. E. Animal Fats and Oils.

—— Vegetable Fats and Oils. London, 1897.

BENEDIKT R. Analyse der Fette und Wachsarten. Berlin, 1892.

BEAUVISAGE, G. Les Matières Gras. Paris, 1891.

BOCKAIRY, L. Huiles Comestibles. Analyse des Matières Alimentaires (Girard et Dupré), p. 401. Paris, 1894.

BORNEMANN, G. Die Fetten Oele des Pflanzen und Thierreiches. Weimar, 1889.

BRANNT, W. T. Animal and Vegetable Fats and Oils. Phila., 1888.

CANNIZZARO, S., and FABRIS, G. Reliability of Tests for Determining the Purity of Olive Oil. Rome, 1891.

GILL, A. A Short Handbook of Oil Analysis. Phila., 1900.

HEHNER, O., and Mitchell, C. A. On the Determination of Stearic Acid in Fats. Analyst, 21, 316.

HOPKINS, E. H. The Oil Chemists' Handbook. New York, 1901.

HUNT, F. W. A Comparison of Methods used to Determine the Iodine Values of Oils. Jour. Soc. Chem. Ind., 1902, 454.

LEWKOWITSCH, J. Chemical Analysis of Oils, Fats, and Waxes. London, 1898.
—— Laboratory Companion to Fats and Oils Industries. London, 1901.
LICHTENBERG, C. Die Fettwaaren und fetten Oele. Weimar, 1880.
LYTHGOE, H. C. Readings on the Zeiss Butyro-refractometer of Edible Oils and Fats. Technology Quarterly, 16, 1903, p. 222.
MACFARLANE, T. Olive Oil. Can. Inl. Rev. Dept. Bul. 67.
RAE & Co. Prima Arborum. The Olive Tree and its Fruit. New York, 1887.
SCHÄDLER, C. Die Untersuchung der Fette, Oele, Wachsarten, etc. Leipzig, 1889.
—— Die Technologie der Fette und Oele des Pflanzen und Thierreichs. Leipzig, 1892.
THALMANN, F. Die Fette und Oele. Leipzig, 1881.
TOLMAN, L. M. Edible Oils and Fats. U. S. Dept. of Agric., Bur. of Chem., Bul. 65, p. 20.
TOLMAN, L. M., and MUNSON, L. S. Use of the Bechi Test in Olive Oils. Jour. Am. Chem. Soc., 24, 1902, 397.
—— Refractive Indices of Salad Oils. Jour. Am. Chem. Soc., 24, 1902, 754.
—— Iodine Absorption of Oils and Fats. Jour. Am. Chem. Soc., 1903, 244.
—— Olive Oil and its Substitutes. U. S. Dept. of Agric., Bur. of Chem., Bul. 77.
VILLON, A. M. Les Corps Gras. Paris, 1890.
VULTE, H. T., and GIBSON, H. W. Nature and Properties of Corn Oil. Jour. Am. Chem. Soc., 23, 1.
WRIGHT, A. C. Analysis of Oils and Allied Substances. New York, 1903.
WRIGHT, C. R. A. Animal and Vegetable Fixed Oils, Fats, Butters, and Waxes. London, 1894.

California Exp. Sta. Buls. 104, 123, 129, 137, et al. on California Olives and Olive Oils.
California Exp. Sta. An. Reports, 1892 et seq.
Connecticut Exp. Sta. An. Report, 1897, p. 44.

REFERENCES ON BUTTER.



GENTH, F. A. The Necessity for a Butter Standard. Penn Dept. of Agric. An. Rep., 1897, p. 549.

GIRARD et BREVANS. Le Margarin et le Beurre Artificiel. Paris, 1889.

HEHNER, O., and ANGELL, A. Butter, its Analysis and Adulteration. London, 1877.

HESS, W. H., and DOOLITTLE, R. E. Methods for the Detection of Process or Renovated Butter. Jour. Am. Chem. Soc., 22, 150.

HUMMEL, J. A. Examination of Brown and Taylor's Official Method of Identifying Butter. Jour. Am. Chem. Soc., 22, 1900, 327, 703.

LANG, V. Fabrikation von Kunstbutter. Leipzig, 1885.

LOW, A. W. Testing for a Yellow Azo-Color in Fats. Jour. Am. Chem. Soc., 20 (1898), p. 889.

MACFARLANE, T. Butter. Can. Inl. Rev. Dept. Bul. 16.

MARTIN, E. W. Detection of Artificial Coloring Matter in Butter, Oleomargarine, etc. Analyst, 12, 1887, p. 70.

MOORE, R. W. A Test for Carrot Color in Butter. Analyst, 11, 1886, 163.

VON RYN, J. J. L. Composition of Dutch Butter. London, 1902.

DE SCHWEINITZ and EMERY. Use of the Calorimeter in Detecting Adulterations of Butter. Jour. Am. Chem. Soc., 18, p. 174.

SELL, E. Ueber Kunstbutter, ihre Herstellung, etc. Berlin, 1886.

STEBBINS, J. H. On the Reichert Figure of Butter.

ZANE, A. J. General Analyse des Beurres. Paris, 1892.

Farmer's Bulletin 12. Nostrums for Increasing the Yield of Butter.
" " 57. Butter Making on the Farm.
" " 92. Pasteurization in Butter Making.
" " 131. Household Tests for the Detection of Oleomargarine and Renovated Butter.

Minnesota Exp. Station Bul. 74. Digestibility and Food Value of Butter.

New York Exp. Sta. (Ithaca), Bul. 118. Butter Increasers.

North Carolina Exp. Sta. Bul. 166. Butter, its Composition, Artificial Imitation, and Adulterants.

Storrs Conn. Exp. Sta. 11th An. Rep., page 85. Bacteriology in Butter Making.

REFERENCES ON LARD.

COCHRAN, C. B. Detection of Foreign Fats in Lard and Butter.

CONROY, M. Lard, its Adulteration with Cottonseed Oil and Detection Thereof. Analyst, 13, 203.

GLADDING, T. S. Examination of Lard for Adulteration. Analyst, 14, 32.

— - Microscopic Detection of Beef Fat in Lard. Jour. Am. Chem. Soc., 18, 189.

HEHNER, O. On Belfield's Test for Beef Stearin in Lard. Analyst, 27, 247.

MACFARLANE, T. Lard. Canadian Inl. Rev. Dept. Bul. 7.

STOCK, W. F. K. On the Estimation of Beef Fat in Lard. Analyst, 19, 2.

TENNILE, G. F. Determination of Solid Fats in Compound Lard. Jour. Am. Chem. Soc., 19, 51.

WESSON, D. Examination of Lard for Impurities. Jour. Am. Chem. Soc., 17, 723.

WILEY, H. W. Quantitative Estimation of Adulterants in Lard. Analyst, 14, 1889, p. 73.
—— Lard and Lard Adulterations. U. S. Dept. of Agric., Div. of Chem., Bul. 13, part 4.
WIRTHLE, F. Detection of Cottonseed Stearin in Lard. Analyst, 27, 247.

Conn. Exp. Sta. An. Rep., 1896, p. 128.
Mass. State Board of Health An. Rep., 1895, p. 668.



CHAPTER XIII.

SUGAR AND SACCHARINE PRODUCTS.

Nature and Classification of Sugars.—Of all classes of food materials the sugars from their great solubility are the most readily available, and on this account are very valuable as nutrients. As in the natural processes of digestion the starches and more difficultly digestible of the carbohydrates are converted into sugar and thus rendered assimilable, so by processes quite analogous to those that take place in the alimentary tract, the chemist converts these same carbohydrates into sugar as an end-point for purposes of definite determination.

The sugars are characterized by their sweet taste, their ready solubility in water, their power to rotate the plane of polarized light, and their insolubility in ether and absolute alcohol.

The sugars occurring commonly in food naturally divide themselves into two groups: First, the Saccharoses, or cane sugar group, having the composition $C_{12}H_{22}O_{11}$, of which the most prominent members are sucrose, maltose, and lactose; and, second, the Glucoses, or grape sugar group, expressed by the formula $C_6H_{12}O_6$, which includes dextrose, levulose, and galactose, besides other less common sugars.

The members of both groups are intimately related. Thus by the ordinary process of so-called inversion sucrose, or cane sugar, belonging to group 1, is converted by the action of heat and dilute acid into two sugars, dextrose and levulose, which are members of group 2, in accordance with the following reaction:

$$\underset{\text{Cane sugar}}{C_{12}H_{22}O_{11}} + H_2O = \underset{\text{Dextrose}}{C_6H_{12}O_6} + \underset{\text{Levulose}}{C_6H_{12}O_6}.$$

The same formula expresses also the result that takes place when lactose, or milk sugar, is heated with dilute acids, breaking up into dextrose and galactose.

Occurrence.—Sugars occur in roots, grasses, stems of plants, trunks of trees, leaves, and fruits, usually in the form of cane sugar, or sucrose, and of invert sugar (dextrose and levulose) mixed in varying proportions.

The following table from Buignet * shows the kind and amount of sugars occurring in some of the common fruits:

	Cane Sugar.	Reducing Sugar.	Acid.
Apricots	6.04	2.74	1.864
Pineapples	11.33	1.98	.547
English cherries	.00	10.00	.661
Lemons	.41	1.06	4.706
Figs	.00	11.55	.057
Strawberries	6.33	4.98	.550
Raspberries	2.01	5.22	1.380
Gooseberries	.00	6.40	1.574
Oranges	4.22	4.36	.448
Peaches (green)	.92	1.07	3.900
Pears (Madeleine)	.36	8.42	.115
Apples	5.28	8.72	1.148
"	2.19	5.45	.633
Prunes	5.24	2.43	1.288
Grapes (hothouse)	.00	17.26	.345
" green	.00	1.60	2.485

CANE SUGAR, OR SUCROSE.

Nature and Occurrence.—This, the most common of all the sugars, is nearly always understood by the unqualified term of sugar. It crystallizes in monoclinic prisms. Its specific gravity is 1.595. Its melting-point is about 160° C. Its specific rotary power $[\alpha]_D$ in solutions having a concentration of from 10 to 20 grams in 100 cc. is, according to Tollens,



The Sugar Cane (*Saccharum officinarum*) is cultivated principally in Louisiana and other southern states, in Cuba and the West Indies, and in the Hawaiian Islands. Its growth and cultivation form an industry in nearly all tropical countries.

Allen * has compiled the following table showing the composition of the juice of the sugar cane from different localities:

Locality and Kind of Cane.	Water.	Sugar.	Woody Fiber.	Salts.	Authority.
Martinique.....	72.1	18.0	9.9	.....	Peligot
Guadaloupe.....	72.0	17.8	9.8	0.4	Dupuy
Havana.....	77.0	12.0	11.0	....	Casaseca
Cuba.....	65.9	17.7	16.4	....	Casaseca
Mauritius.....	69.0	20.0	10.0	1.0	Icery
Ribbon cane.....	76.73	13.39	9.07	.39	Avequin
Tahiti.....	76.08	14.28	8.87	.35	Avequin

The composition of raw cane sugar ash according to Monier is as follows:

RAW CANE SUGAR ASH.

Carbonate of calcium.....	49.00
" " potassium.....	16.50
Sodium and potassium sulphate.....	16.00
Sodium chloride.....	9.00
Silica and alumina.....	9.50
	100.00

Manufacture of Cane Sugar.—The process of manufacturing raw sugar from sugar cane is briefly as follows: The juice is first extracted from the canes by crushing in roll mills and is freed from nitrogenous bodies, organic acids, etc., by the process of *defecation*, which consists in heating to coagulate the albumin, and nearly neutralizing with milk of lime, the impurities being removed as a scum. The juice is then subjected to evaporation and crystallization, the raw, or muscovado sugar, which contains from 87 to 91 per cent of sucrose, being separated from the molasses, which is the mother liquor, by draining or by centrifugal.

Some of the best grade of muscovado, or raw sugar, is used as "brown sugar" without further refining, and much of the molasses is used as a table syrup and for cooking, while the lower grades of molasses are used in the manufacture of rum.

* Com. Org. Analysis, 1, p. 315.

The following table from Thorpe * shows the average composition of raw and refined sugar:

	Cane Sugar.	Glucose[1].	Water.	Organic Matter.	Ash.
RAW SUGAR.					
Good centrifugal	96.5	0.75	1.50	0.85	0.40
Poor centrifugal	92.0	2.50	3.00	1.75	0.75
Good muscovado	91.0	2.25	5.00	1.10	0.65
Poor muscovado	82.0	7.00	6.00	3.50	1.50
Molasses sugar	85.0	3.00	5.00	5.00	2.00
Jaggary sugar	75.0	11.00	8.00	4.00	2.00
Manilla sugar	87.0	5.50	4.00	2.25	1.25
Beet sugar, 1st	95.0	0.00	2.00	1.75	1.25
Beet sugar, 2d	91.0	0.25	3.00	3.25	2.50
REFINED SUGAR.					
Granulated sugar	99.8	0.20	0.00	0.00	0.00
White coffee sugar	91.0	2.40	5.50	0.80	0.30
Yellow X C sugar.	87.0	4.50	6.00	1.50	1.00
Yellow sugar	82.0	7.50	6.00	2.50	2.00
Barrel sugar.	40.0	25.00	20.00	10.00	5.00

[1] The term "glucose" includes sugars which reduce Fehling's solution, but are not necessarily optically active.

The following minimum and maximum figures are taken from analyses made by Babington † of twenty-two samples of brown sugar and thirty-one samples of molasses.

BROWN SUGAR.

Direct polarization	84	to	87
Invert "	−27	"	−29
Sucrose by Clerget	83.5	"	91.5
Reducing sugar	3	"	6

The Sugar Beet (*Beta vulgaris*) is grown chiefly in France and Germany, and to a lesser extent in Holland and England. The successful growth of the sugar beet in the United States is confined mainly to California, Utah, and Nebraska, and the entire output of beet sugar in this country is comparatively small.

According to R. Hoffmann, sugar beets have about the following composition, three types being selected—first, those poor in sugar; second, those having a medium sugar content, and third, those rich in sugar:

COMPOSITION OF THE SUGAR BEET.

	First Type.	Second Type.	Third Type.
Water	89.20	83.20	75.20
Sugar	4.00	9.42	15.00
Nitrogenous compounds	1.00	1.64	2.20
Non-nitrogenous compounds:			
Soluble	4.13	3.34	4.23
Insoluble (cellulose)	1.01	1.50	2.07
Ash	0.66	0.90	1.30
	100.00	100.00	100.00

The following is the mean composition of ten samples of California sugar beet:*

Per cent juice extracted	61.38
Specific gravity	1.062 to 1.075
Per cent of reducing sugar	0.91
Per cent of sucrose	14.38
Total solids calculated	16.58
Total solids weighed	17.20
Per cent of ash	0.994

The composition of beet sugar ash according to Monier is as follows:

RAW BEET SUGAR ASH.

Carbonates of potassium and sodium	82.20
Carbonate of calcium	6.70
Potassium and sodium sulphate and sodium chloride	11.10
	100.00

Manufacture of Beet Sugar.—In making raw sugar from sugar beets the latter are first washed and sliced by machinery and the juice extracted

* U. S. Dept. of Agric., Div. of Chem., Bul. 27, p. 202.

by diffusion or digestion with warm water. The juice is then clarified or defecated in much the same manner as that from the sugar cane, after which it is usually bleached with sulphur dioxide.

The subsequent evaporation and crystallization are carried out usually in vacuum pans, and the sugar separated out by centrifugals.

Beet sugar molasses is unfit for food, due to the presence of nitrogenous bodies, which give it a very unpleasant taste and smell.

Process of Refining.—In refining raw sugar, a syrup is made, which is subjected to centrifuging and further defecation, using lime, clay, liquid blood, calcium acid phosphate, and other substances as clarifiers. The juices are then filtered, first through cloth bags and then through bone char, after which they are evaporated and allowed to crystallize, the resulting granulated sugar being separated, as in the case of raw sugar, by centrifugal machines.

Granulated Sugar of commerce is without doubt the purest food product on the market, being generally 99.8% sucrose. It is usually treated with an extremely weak solution of ultramarine to counteract the natural yellow color.

The syrup from which the granulated sugar is separated forms the "golden," or "drip," syrup used on the table. Its typical composition is as follows: Sucrose, 40%; reducing sugars, 25%; water, 20%; organic matter, 10%; ash, 5%.

The dry sugars, whether white or brown, are rarely subjected to adulteration.

Maple Sap.—The sap of the maple tree, *Acer saccharinum*, or *Acer barbatum*, furnishes a sugar considerably prized for its peculiar flavor. The maple sugar industry is largely confined to the northeastern states



with great care, since this contains nitrogenous matters that would cause fermentation in the finished product. Pure cane sugar is never commercially produced from the maple sap, since the refining process would deprive it of the flavor which gives to maple sugar the chief value.

McGill gives the following as the average analyses of six samples of maple syrup of known purity:

Saccharimeter Direct.	Saccharimeter Invert.	Cane Sugar by Clerget.	By Copper.		Ash.	Water.	Solids.
			Reducing Sugar.	Cane Sugar.			
+62.2	−21.2	62.4	.42	63.36	.53	35.70	64.30

The following is a summary of the analyses of twenty samples of maple sugar:*

Sucrose	71.80 to 86.89%
Reducing sugar	Trace " 12.19%
Water	6.77 " 11.57%
Ash	0.31 " 1.50%

Methods for the analysis of maple sugar are identical with those for raw, or brown, sugar.

Adulteration of Maple Sugar and Syrup.—The chief adulterants of maple sugar are brown, or molasses sugar, and white, or refined sugar, the latter being often used in mixture with burnt or inferior maple stock, which itself would be abnormally dark in color and of a rank taste. Maple syrup is commonly adulterated with golden, or drip syrup, with commercial glucose, with molasses, and with refined sugar.

The ash of pure maple syrup, according to Sharples, should not be less than 0.35 or 0.40 per cent, a lower ash indicating the admixture of refined cane sugar. On the other hand, the addition of molasses, or brown sugar stock would tend to raise the ash far above the average for pure maple, as well as to increase the reducing sugars. A high reducing sugar, say over 5%, while suspicious, should not be assumed as conclusive evidence in itself of molasses sugar, since old and inferior samples of maple sugar and syrup are sometimes found with reducing sugar abnormally high. It is frequently possible to identify brown, or molasses, sugar, especially when it forms the larger portion of the alleged maple sugar or syrup by the physical sense of taste. When the perfectly charac-

* U. S. Dept. of Agric., Div. of Chem., Bul. 5, p. 198.

teristic taste of brown, or molasses sugar, or of "drip syrup," so far predominates over the maple flavor as to be unmistakable, especially in cases where the maple flavor is entirely lacking, one need have little hesitation in condemning the product.* See also Appendix, p. 767.

Sorghum (*Andropogon sorghum*, variety *saccharatus*) has for many years been grown quite extensively in the southern and western states, and used as a source of syrup, but only in recent years has it been found practicable to produce crystallized cane sugar from it on account of the presence of starch, uncrystallizable sugar, etc.

Much experimental work has been done of late along this line by the U. S. Department of Agriculture. The sorghum plant is as yet, however, a very small factor in the production of cane sugar, though much progress is being made.

The composition of the juice of the sorghum plant is shown by the following results of analyses of eleven varieties made by Hardin: †

Total solids	15.97	to 18.71
Specific gravity	1.0656	" 1.0775
Solids not sugar	5.02	" 10.63
Cane sugar	2.81	" 8.01
Reducing sugars	3.87	" 7.55

Some varieties of sorghum juice have been known to contain 15 or even 17 per cent of sucrose.

In making syrup from sorghum, the ripe canes are crushed, the juice is heated with milk of lime, and the scum removed. The juice is then concentrated usually in open pans to the required consistency.



The specific rotary power of dextrose is

$$[\alpha]_D = 52.3, \quad [\alpha]_j = 58.$$

A normal solution of dextrose on the Soleil-Ventzke scale polarizes at 78.6°. For the commercial preparation of dextrose see p. 471.

U. S. Standards for Various Sugars.—*Standard* 70 *sugar*, or *brewers' sugar*, is hydrous starch sugar containing not less than 70% of dextrose, and not more than 0.8% of ash.

Standard 80 *sugar, climax*, or *acme sugar*, is hydrous starch sugar containing not less than 80% of dextrose, and not more than 1.5% of ash.

Standard anhydrous grape sugar is anhydrous grape sugar containing not less than 95% of dextrose without water of crystallization, and not more than 0.8% of ash.

The ash of these standard products consists almost entirely of chlorides and sulphates of lime and soda.

LEVULOSE.

Levulose is of importance in foods only as the product of inversion of cane sugar. It is prepared by the action of dilute acids on inulin. Normally it is in the form of a syrup, being with difficulty crystallized, but with extreme care pure anhydrous levulose can be obtained. Diabetene is a commercial form of dry levulose. Levulose is formed with dextrose in the inversion of cane sugar (p. 461), and with dextrose occurs in honey and in many fruits. The specific rotary power of levulose varies with the temperature. At 15° C. $[\alpha]_D = -98.8°$, decreasing by 0.6385° for each degree increase in temperature. Its left-handed reading on the Ventzke sugar scale at 15° C. is equivalent to 148.6°. Levulose is sweeter than dextrose. Its reducing power on Fehling's solution is assumed to be the same as that of dextrose.

MALT SUGAR, OR MALTOSE.

Maltose ($C_{12}H_{22}O_{11} + H_2O$) is of little importance from the standpoint of the food analyst, excepting as an ingredient of commercial glucose, and as being the sugar produced by the action of ptyaline, the ferment of the saliva on the starch of food in the ordinary process of digestion. When gelatinized starch is subjected to treatment with malt extract at 55° to 60° C., it is converted into dextrin and maltose as follows:

$$\underset{\text{Starch}}{C_{18}H_{30}O_{15}} + H_2O = \underset{\text{Dextrin}}{C_6H_{10}O_5} + \underset{\text{Maltose}}{C_{12}H_{22}O_{11}}.$$

In its commercial preparation maltose is separated from dextrin by crystallization in alcohol. By the action of weak acids and heat both dextrin and maltose are further converted into dextrose.

Maltose usually crystallizes in minute needles, and its molecule of water is expelled at 110° C. It is somewhat less soluble in water than dextrose. It is slightly soluble in alcohol, though less than sucrose. Solutions of maltose possess the property of birotation; i.e., when freshly prepared they do not at once assume their true optical activity. The rotation of a freshly prepared solution of maltose increases on standing, requiring several hours to reach its maximum. The specific rotary power, according to O'Sullivan, of anhydrous maltose is $[\alpha]_D = 139.2$, $[\alpha]_j = 154.5$. For hydrated maltose $[\alpha]_D$ would thus be 132.2.

A normal solution of maltose on the Soleil-Ventzke scale should polarize at 198.8°.

DEXTRIN. COMMERCIAL GLUCOSE.

DEXTRIN, $(C_6H_{10}O_5)_n$, possesses more the nature of a gum than of a sugar, and is sometimes called British gum. It is said to occur naturally in the sap of various plants, but this is not definitely assured.

It undoubtedly occurs in beer and in bread crust, and is one of the constituents of commercial glucose. Like starch, it is convertible by hydrolysis with acid into dextrose. By treatment of starch with malt extract or diastase, starch is converted into dextrin and maltose, these two bodies being separated, in the commercial preparations of dextrin, by repeated treatment with alcohol.

Dextrin is an uncrystallized, colorless, tasteless body, capable of being pulverized. It is readily soluble in water, slightly soluble in dilute alcohol, but insoluble in alcohol of 60% or stronger. It is not colored



juice is filtered through bone black, and finally concentrated by evaporation, the degree of conversion and of concentration depending on whether the liquid glucose or the solid dextrose is wanted for the final product. The end product obtained by complete conversion is the dry commercial grape sugar, or dextrose, which is purified by repeated crystallization.

Commercial glucose is a mixture of dextrin, maltose, and dextrose of the following varying composition:

Dextrin	29.8%	to 45.3%
Maltose	4.6%	" 19.3%
Dextrose	34.3%	" 36.5%
Ash	0.32%	" 0.52%
Water	14.2%	" 17.2%

Calcium sulphate is usually found in the ash.

Solid commercial grape sugar, or dextrose, has the following composition:

Dextrin	0%	9.1%
Maltose	0%	1.8%
Dextrose	72%	99.4%
Ash	0.3%	0.75%
Water	0.6%	17.5%

U. S. Standard glucose, mixing glucose, or *confectioners' glucose,* is colorless glucose, varying in density between 41° and 45° Baumé, at a temperature of 100° F. (37.7° C.). It conforms in density, within these limits, to the degree Baumé it is claimed to show, and for a density of 41° Baumé contains not more than 21% of water, and for a density of 45° not more than 14%. It contains on a basis of 41° Baumé not more than 1% of ash, consisting chiefly of chlorides and sulphates of lime and soda.

Healthfulness of Glucose.—The analyst alleging commercial glucose as an adulterant is frequently asked in court as to its healthfulness, so that the following conclusions of a committee appointed some years ago by the National Academy of Sciences to ascertain among other things whether there is any danger attending the use of this product in food are in point: "First, that the manufacture of sugar from starch is a long-established industry, scientifically valuable and commercially important; second, that the processes which it employs at the present time are unobjectionable in their character and leave the product uncontaminated; third, that the starch sugar thus made and sent into commerce is of excep-

tional purity and uniformity of composition and contains no injurious substances; and fourth, that though having at best only about two-thirds the sweetening power of cane sugar, yet starch sugar is in no way inferior in healthfulness, there being no evidence before the committee that maize starch sugar, either in its normal condition or fermented, has any deleterious effect upon the system, even when taken in large quantities."

MILK SUGAR, OR LACTOSE.

Lactose ($C_{12}H_{22}O_{11}+H_2O$) is prepared commercially from skim-milk by coagulating with rennet and digesting the whey with chalk and aluminum hydroxide. The insoluble matter is filtered out, and the filtrate is concentrated *in vacuo* to a syrup, which, on standing, yields crystals of lactose. The product is purified by repeated crystallization.

Lactose ordinarily crystallizes in rhombic, hemihedral crystals. Its specific gravity is 1.525. Its water of crystallization is lost by drying at 130° C. It is soluble in 6 parts of cold water, and in 2½ or less of boiling water. It is insoluble in absolute alcohol and ether. It has a very slightly sweet taste.

The specific rotary power of milk sugar, after remaining in solution long enough to overcome its birotation, is

$$[\alpha]_D = 52.5.$$

In the ordinary souring of milk the lactose becomes converted into lactic acid.

On heating lactose with dilute acids it undergoes inversion, forming dextrose and galactose in accordance with the formula given on p. 461, illustrating the inversion of cane sugar.



Raffinose occurs in beet root molasses to the extent of from 3 to 4 per cent. It is a crystalline, slightly sweet substance, soluble in water and slightly soluble in alcohol. It does not reduce Fehling's solution, but readily undergoes fermentation. On inversion it splits up into levulose and melibiose ($C_{12}H_{22}O_{11}$).

The melting-point of raffinose is 118° to 119° C. Its specific rotary power $[\alpha]_D = +104.5$ at a temperature of 20° C.

THE POLARISCOPE AND SACCHARIMETRY.

A full discussion of the principles of polarized light and even a detailed description of their application to the polariscope will not be given here, but the reader who wishes full information along this line is referred to the various text-books, and especially to those of Tucker, Spencer, and Landolt,* in which various forms of polariscopes are described and their underlying principles discussed.

The Soleil-Ventzke Saccharimeter is the one most commonly used in this country, being adopted as the standard for all United States government work. Fig. 99 shows this instrument, known as the half-shadow

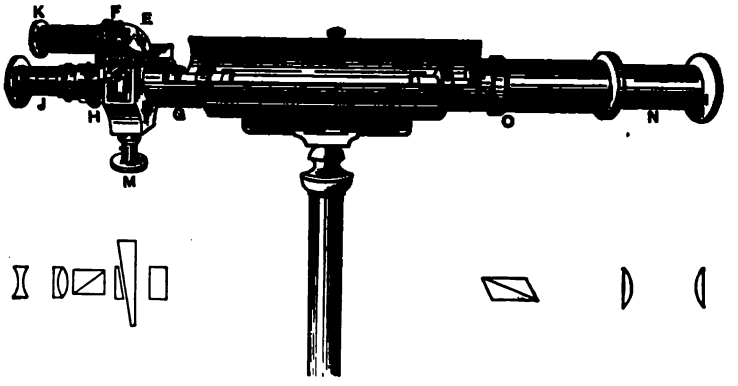


FIG. 99.—Single-wedge Saccharimeter.

apparatus, in its simplest form with a single movable wedge in its compensating system.

An excellent light for work with this instrument is that furnished by the Welsbach burner, a convenient form of lamp being shown in Fig. 110, in which the burner is inclosed in a sheet-metal chimney of suitable construction. An argand, gas, or kerosene burner may however be used,

* See references, p. 522.

and in a late form of Schmidt and Haensch instrument, Fig. 100, a specially constructed incandescent electric lamp is supplied.

The Single-wedge Saccharimeter.—The following description of the saccharimeter and directions for its use are from the revised regulations of the U. S. Internal Revenue Department. The tube *N*, Fig. 99, contains the illuminating system of lenses and is placed next to the lamp; the polarizing prism is at *O* and the analyzing prism at *H*. The quartz wedge compensating system is contained in the portions of the tube marked *FEG* and is controlled by the milled head *M*. The tube *J* carries a small telescope, through which the field of the instrument is viewed, and just above is the reading-tube *K*, which is provided with a mirror and magnifying lens for reading the scale.

The tube containing the sugar solution is shown in position in the trough between the two ends of the instrument. In using the instrument the lamp is placed at a distance of at least 200 mm. from the polarizing end; the observer seats himself at the opposite end in such a manner as to bring his eye in line with the tube *J*. The telescope is moved in or out until the proper focus is secured to give a clearly defined image, when the field of the instrument will appear as a round, luminous disk, divided into halves by a vertical line passing through its center, and darker on one half of the disk than on the other, when the compensating quartz wedge is displaced from the neutral position. If the observer, still looking through the telescope, will now grasp the milled head *M* and rotate it first one way and then the other, he will find that the appearance of the field changes, and at a certain point the dark half becomes light and the light half dark. By rotating the milled head delicately backward and forward over this point he will be able to



With the milled head set at the point which gives the appearance of the middle disk shown in Fig. 103, the eye of the observer is raised to the reading tube *K*, which is adjusted to secure a plain reading of the divisions, and the position of the scale is noted. It will be seen that the scale proper is attached to the quartz wedge, which is moved by the milled head; and attached to the other quartz wedge is a small scale called a *vernier*, which is fixed, and which serves for the exact determination of the position of the movable scale with reference to it. On each side of the zero line of the vernier a space corresponding to *nine* divisions of the movable scale is divided into *ten* equal parts. By this device the fractional part of a degree indicated by the position of the zero line is ascertained in

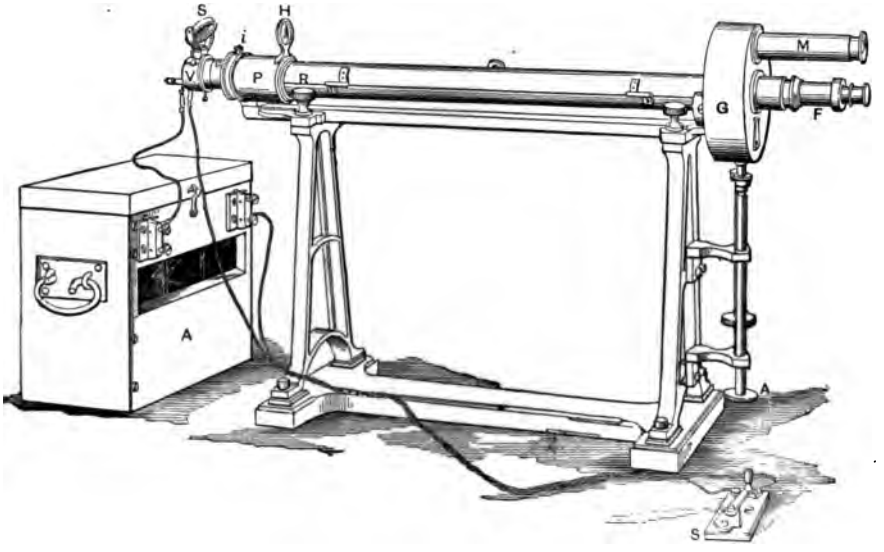


FIG. 100.—Single-wedge Soleil-Ventzke Saccharimeter, mounted on Bock Stand and provided with Incandescent Electric Lamp.

tenths; it is only necessary to count from zero until a line is found which makes a continuous line with one on the movable scale.

With the neutral field, as indicated above, the zero of the movable scale should correspond closely with the zero of the vernier, unless the zero point is out of adjustment.

Adjusting the Instrument.—If the observer desires to secure an *exact* adjustment of the zero of the scale, or in any case if the latter deviates more than one-half of a degree, the zero lines are made to coincide by moving the milled head and securing a neutral field at this point by

means of the small key which comes with the instrument, and which fits a small nipple on the *left*-hand side of *F*, the fixed quartz wedge of the compensating system. This nipple must not be confounded with a similar nipple on the *right*-hand side of the analyzing prism *H*, which it fits as well, but which *must never be touched*, as the adjustment of the instrument would be seriously disturbed by moving it. With the key on the proper nipple it is turned one way or the other until the field is neutral. Unless the deviation of the zero be greater than 0.5° it will not be necessary to use the key, but only to note the amount of the deviation, and for this purpose the observer must not be content with a single setting, but must perform the operation five or six times and take the mean of these different readings. If one or more of the readings show a deviation of more than 0.2° from the general average they should be rejected as incorrect. Between each observation the eye should be allowed a moment of rest.

The Scale usually has 110 equal divisions on one side of the zero for reading right-handed polarization, and 20 equal divisions on the other side for left-handed polarization. The scale is an arbitrary one, based on the plan that a normal aqueous solution of pure cane sugar (26.048 grams made up to 100 cc.) will read exactly 100° or divisions to the right of the zero.

The accuracy of various portions of the scale may be verified by quartz control plates of varying thickness, usually mounted in tubes, the correct polariscopic reading of each of which plates has been accurately determined, this reading being as a rule marked on the tube. As the sugar value of such a quartz plate varies with the temperature, the temperature at which the particular reading marked thereon applies is usually specified, and in many cases a table giving its exact value at



FIG. 103.—Appearances of the Field in the Half-shadow Saccharimeter.



FIG. 104.—Appearances of the Field in the Triple-shade Saccharimeter.



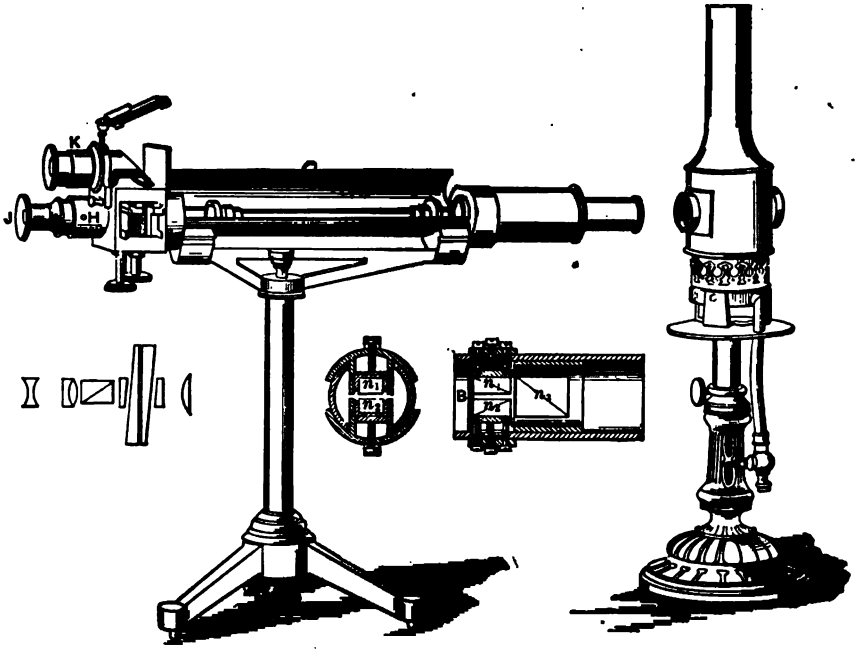


FIG. 101.—Double-wedge, Triple-shade Soleil-Ventzke Saccharimeter.

is the triple-shadow instrument, the construction of the polarizer being shown in Fig. 102.

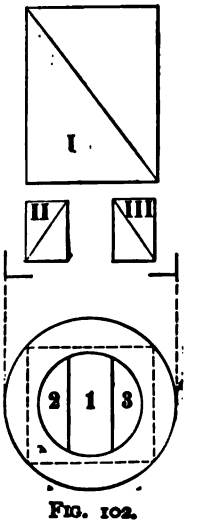


FIG. 102.

In this form the analyzer is the same as in the foregoing instruments, but the polarizer consists of one large and two small Nicol prisms I, II, and III, the construction and arrangement being such that when the compensating wedges are at the neutral point, sections 1, 2, and 3 of the circular field (corresponding respectively to the prisms I, II, and III) are evenly lighted, forming a circular uniformly colored field, while in any other position of the wedges section 1 is dark while 2 and 3 are light or vice versa. The accompanying diagram, Fig. 104, shows the appearance of the field of this instrument in the three positions of the quartz wedge, viz., at the neutral point and at both sides thereof.

The lamp used for illumination should be separated from the polariscope on account of the influence of its heat on the readings. This is best accomplished by having the lamp in a separate compartment from the polariscope, so

that both are on opposite sides of a partition, an opening in which transmits the light. In any event some kind of screen should be interposed between the two. Best results are obtained if the room in which the observations are made is dark.

Comparisons of Scales of Various Polariscopes.—Besides the Soleil-Ventzke instrument, there are various other forms of polariscope. Among the best known of these are Laurent's, Wild's, and Duboscq's, all of which are made with scales reading in circular degrees, while in some cases modified forms have scales in which, like the Soleil-Ventzke, percentages of sugar are directly read off. Some instruments are provided with double scales reading both circular degrees and percentages of sugar, and in certain of the Duboscq instruments additional scales for percentages of milk sugar and diabetic sugar are provided.

In the Wild, Duboscq, and Laurent instruments the source of light is the sodium flame, yielding what is termed a monochromatic light. This is produced by fused sodium chloride passing through a Bunsen flame, various mechanical devices being employed for making the light continuous. In the Ventzke instrument, as was stated above, the ordinary light from a bright gas or oil flame is used.

For convenience in conversion of readings on one instrument to their equivalents on other scales, the following factors can be used:

1° Ventzke	=0.3468°	angular rotation *D*.
1° angular rotation *D*	=2.8835°	Ventzke.
1° Ventzke	=2.6048°	Wild (sugar scale).
1° Wild (sugar scale)	=0.3840°	Ventzke.
1° " " "	=0.1331°	angular rotation *D*.
1° angular rotation *D*	=0.7511°	Wild (sugar scale)
1° Laurent (sugar scale)	=0.2167°	angular rotation *D*.
1° angular rotation *D*	=4.6154°	Laurent (sugar scale).
1° Soleil-Duboscq	=0.2167°	angular rotation *D*.
1° " "	=0.2159°	" " "

Specific Rotary Power.—This is a theoretical term to express a standard by which the various optically active substances may be compared, and is understood to mean the amount in angular degrees through which the plane of polarization of a ray of light of stated wave length is rotated by 1 gram of a given substance in aqueous solution of 1 cc. and forming a column 1 decimeter in length. The actual rotary power of a solution varies directly with the length of the column traversed by the light, with the concentration of the solution, and with the wave length of light, hence the need of a purely theoretical basis for purposes of comparison.

The specific rotary power is usually expressed as $[\alpha]_D$ or $[\alpha]_j$, the letters D or j indicating the character of the light. Thus, D indicates the monochromatic light obtained from the sodium flame, named from the D line of Fraunhofer in the yellow portion of the spectrum, while j (from the French *jaune*) indicates what is known as the *transition tint*, the rose-purple color produced when ordinary white light passes through the polarizer and analyzer, placed with their principal sections parallel to each other and with a plate of quartz 3.75 mm. thick interposed between them.*

The specific rotary power is determined as follows:

$$[\alpha]_D \text{ or } [\alpha]_j = \frac{100a}{cl},$$

where a = observed angular rotation,

c = grams of the substance in 100 cc. of the solution, and

l = length of the observation-tube in decimeters; or, in cases where, instead of the grams per 100 cc., the percentage composition is known (expressed by p = grams of the substance in 100 grams of the solvent), and the specific gravity (expressed by d), then $[\alpha]_D$ or $[\alpha]_j = \frac{100a}{pdl}$.

Birotation.—In polarizing solutions of all the common sugars other than sucrose the phenomenon of birotation should be taken into account, whereby a change in optical activity is shown by standing. Thus, solutions of dextrose, levulose, and lactose polarize much higher when freshly prepared than after long standing, requiring in some instances several hours before the lowest or normal figure is reached. Maltose, on the other hand, increases in polarization after standing in solution. By

* Some confusion is caused by the adoption of the characters D and j, since both would naturally seem to indicate yellow light. The so-called *transition tint* above defined is, however, complementary to the mean yellow, or *jaune moyen*, and it is the complementary color and not the yellow itself that is indicated by the character j

boiling the solution it may be at once brought to its correct reading. The desired result may also be accomplished by adding a few drops of ammonia, either treatment being resorted to before the solution is made up to the required volume.

ANALYSIS OF CANE SUGAR AND ITS PRODUCTS.

Qualitative Tests for Sucrose.—(*a*) *Polariscope Test.*—The substance to be tested, if not already in solution, is dissolved in water, and if the solution is not perfectly clear, is clarified by the addition of alumina cream or by subacetate of lead (p. 481) and filtered. An observation tube is filled with the clear solution and the polariscope reading noted. A measured portion of the same solution is then treated with one-tenth its volume of concentrated hydrochloric acid and is subjected to inversion (p. 483), after which the same tube as before is filled with the inverted solution and a second reading obtained, one-tenth of the observed reading being added for the true invert polariscopic reading. If the two readings are virtually the same, sucrose is absent, but, in the presence of sucrose, the second reading will be considerably lower than the first or may even be to the left of the zero.

(*b*) *Test with Nitrate of Cobalt.**—Prepare a 5% solution of cobaltous nitrate, and a 50% solution of potassium hydroxide. If the sugar solution to be tested contains dextrin or gums, these should first be removed by treatment with alcohol. 15 cc. of the sugar solution to be tested are mixed with 5 cc. of the cobaltous nitrate reagent, and 2 cc. of the potassium hydroxide solution are added. Sucrose produces under these conditions a permanent amethyst-blue color, while dextrose gives at first a turquoise-blue passing over into light green. In a mixture of the two



Determination of Moisture.—5 grams of the sample a
weighed in a flat, tared platinum dish, and dried to constar
vacuo, or in a McGill oven* in a current of air, at a tem
exceeding 70° C. Formerly it was customary to dry sugars
it has recently been shown that at a temperature in excess of 75° 1
levulose becomes dehydrated.

Determination of the Ash.—The residue from the moisture determination is burned slowly and cautiously over a low flame until frothing has ceased. Afterwards increase the flame and ignite to a white ash at a low, red heat.

In igniting saccharine substances which contain an appreciable amount of cane sugar, the contents of the dish will swell up and froth, unless great care be taken, to such an extent as to flow over the sides of the dish, occasioning loss and inconvenience. Such frothing may be largely held in check by directing the flame at first down from above upon the pasty mass, instead of from under the dish as ordinarily, till all is reduced to a dry char, afterwards continuing the ignition from below in the usual manner.

Organic Non-sugars.—These consist mainly of compounds of o
acids, together with gum, coloring matter, albuminous bodies, etc.
are determined by difference between 100% and the sum of the sucrose, invert sugar, moisture, and ash.

Quotient of Purity.—By this term is meant the percentage of pure sugar in the dry substance. It is calculated by dividing the per cent of sucrose by the percentage of total solids and multiplying the result by 100.

Determination of Sucrose by the Polariscope.—Reagents.—*Subacetate of Lead Solution.*†—This is prepared by boiling for half an hour 430 grams of normal lead acetate, 130 grams of litharge, and 1000 cc. of

* A. McGill, Laboratory of Inland Revenue, Ottawa, Canada, has devised a forced-draft water-oven for drying at temperatures between 60° and 90° C. The oven is heated by means of ordinary gas-burners, and the temperature is controlled by introducing at the bottom of the oven a blast of air from a blower run by a small water-motor. Before discharging into the oven, the air-tube enters the water-chamber and is coiled a number of times in order to sufficiently warm the air before it enters the oven. The exit end of the air-tube is covered with a concavo-convex disk in order to distribute the blast and to prevent harmful currents. By regulating the burners and the flow of air, a fairly constant temperature can be obtained. The bottom of the oven is curved instead of flat, to prevent bumping when the water is boiling; a perforated plate serves as a false bottom.

† U. S. P. lead subacetate may be used. This is sometimes sold under the name of Goulard's extract.

water. The mixture is allowed to cool and settle, when the supernatant liquid is diluted to 1.25 specific gravity with recently boiled water.

Alumina Cream is prepared by dividing a cold, saturated, aqueous solution of alum into two unequal portions, to the larger of which add a slight excess of ammonia. Then add by degrees the remaining portion to a faint acid reaction.

Process.—If the Soleil-Ventzke polariscope is to be used, weigh out 26.048 grams of the sugar, which may conveniently be done in the German-silver, tared tray especially designed for this purpose, and which accompanies the Schmidt and Haensch polariscope, Fig. 105. If any other instrument than



FIG. 105.—German-silver Sugar-tray with Tare.

the Soleil-Ventzke is employed, weigh out the standard or normal weight for that instrument (see p. 478). Transfer the sugar by washing to a 100-cc. graduated sugar-flask, and if the solution is perfectly clear, as would be the case with a refined sugar, make up to the mark and shake to insure a uniform solution. If the solution is slightly turbid, or more or less opaque or dark-colored, a clarifier must be added before making up to the mark to obtain a clear solution for polarization. The kind



For additional details as to clarification see page 502, under Molasses.

After adding the clarifier, the flask is filled to the mark with water and shaken, the solution being poured upon a dry filter and the first few cubic centimeters of the filtrate rejected. A 200-mm. observation-tube is filled with the clear sugar solution and the polarization noted. If sucrose is the only optically active substance present, the direct reading on the polariscope will indicate its percentage.

Process of Inversion.—In the presence of invert or other sugars the normal solution as above prepared is subjected to inversion as follows: Take 50 cc. of the above clear sugar solution or of the clarified filtrate, and add 5 cc. of concentrated hydrochloric acid. For this purpose a flask graduated both to 50 and 55 cc. is convenient. Place the flask containing this mixture in water and heat the water gradually till a thermometer in the flask indicates 68° C., maintaining it at between 68° and 70° for five minutes, after which remove the flask and let it cool to room temperature. Filter if necessary, fill a 220-mm. observation-tube with the clear solution, and take the second or invert reading. Or if a 220-mm. tube is not available, use the same tube as before (200 mm.) and add 10% to the reading. Note the temperature at which the invert reading is taken.

The sucrose is obtained by the following formula of Clerget, based on the rotation of cane sugar before and after inversion,

$$S=\frac{100\,(a-b)}{144-\frac{t}{2}},$$

where S = per cent of sucrose, a = direct polarization, b = invert polarization, and t = temperature. Note that if the direct polarization is to the right or positive, and the invert to the left or negative, then $a-b$ would be the sum of the two polarizations.

In many cases where it is almost impossible to obtain a colorless solution for polarization in the 200-mm. tube, a 100-mm. tube may be employed, and the readings multiplied by 2, or half the normal weight,* viz., 13.024 grams, of the sample may be taken and made up to 100 cc., the 200-mm. tube employed, and the readings multiplied by 2.

* Wherever the term "normal weight" occurs hereafter will be meant, unless otherwise noted, the normal weight of sugar for the Soleil-Ventzke polariscope, viz., 26.048, and by a "normal solution" will be meant 26.048 grams in 100 cc. of water. Clerget's formula, as originally worked out by him, was not based on this normal weight, but on 16.35 grams. It is, however, applicable to 26.048 grams.

The determination of sucrose by the Clerget formula is applicable to all mixtures of the common sugars excepting those in which lactose, or milk sugar, is present.

Theory of Inversion.—On p. 461 a reaction is given showing that when sucrose is subjected to inversion by the action of dilute acids or of invertase or yeast it splits up into the two sugars dextrose and levulose, forming equal quantities of each. The dextrose is, however, dextrorotary and the levulose lævorotary. Invert sugar is the term applied to the mixture of dextrose and levulose formed by the inversion of sucrose. The specific rotary power of sucrose varies so little with the temperature as to be regarded for practical purposes as constant. At 87° a solution of invert sugar polarizes at zero. This is due to the fact that the rotary power of levulose, unlike that of sucrose and dextrose, varies with the temperature. At from 87° to 88° the left-handed rotation of the levulose balances the right-handed rotation of the dextrose in the invert sugar, hence the zero reading. As the temperature decreases from 87°, the rotary power of the levulose proportionally increases, till at 0° the normal invert sugar solution would polarize 44° to the left of the zero. On these facts Clerget's formula (p. 483) is based, assuming that a normal solution of pure cane sugar polarizes at +100°, while after inversion the reading for 0° temperature would be −44° and would decrease half a degree for each degree in temperature above 0°. Thus at 20° the invert reading would be −34.

For polariscopes graduated in angular degrees Tuchschmidt* gives the following formula for calculation of sucrose,

$$S = \frac{21.7(a-b)}{\quad},$$

[redacted]

1% solution of methyl blue, and boil over a free flame, noticing particularly the time the solution begins to boil.

If the color disappears in one minute after boiling, there is present at least 0.01% of invert sugar. If it is not completely decolorized by three minutes' boiling, no invert sugar is present.

Determination of Invert Sugar in Cane Sugar Products by the Polariscope.—While invert sugar is best determined by Fehling's solution as described elsewhere, it may be approximately estimated by the polariscope, though less satisfactorily. On p. 508 a method is given for the determination of levulose by polariscopic readings at two different temperatures. Since invert sugar is composed of equal parts by weight of dextrose and levulose, the percentage of levulose multiplied by 2 would give that of invert sugar.

SUGAR DETERMINATION BY COPPER REDUCTION.

Various convenient methods of determining sugars depend on the readiness with which certain of them, known as reducing sugars, act on copper salts, especially on the tartrate of copper, reducing it to cuprous oxide.

This reducing power is exercised in a definite degree under fixed conditions, so that the amount of reducing sugar present may be accurately determined. Of the common sugars, sucrose is the only one that has no direct reducing action, but on undergoing inversion it is converted into reducing sugars, which are readily determined.

Use of Fehling's Solution.—There are various well-known mixtures of copper sulphate, tartaric acid salts (usually Rochelle salts or cream of tartar), and alkalies, called after chemists who have employed them in the determination of the reducing sugars, each one possessing certain advantages, but none have become so widely adopted as Fehling's solution, the use of which in one form or another is now well-nigh universal.

There are a number of methods by which Fehling's solution is employed for this purpose, both volumetric and gravimetric. The former are simpler and quicker of manipulation, and thus are preferable for commercial work where extreme accuracy is not required. The gravimetric methods are usually considered more delicate and accurate, calling for less skill, but more time in arriving at results, and with less of the "personal element" than the volumetric.

Some modifications of the Fehling method, especially as carried out gravimetrically, differ for the various reducing sugars to be determined,

and others are carried out alike, so far as manipulation is concerned, whether the particular sugar to be determined be dextrose, maltose, or lactose.

While, strictly speaking, the reducing power of dextrose, levulose, and invert sugar are not identical, it is customary in commercial work to regard them as such, and no appreciable error arises in consequence except in extreme cases. Thus the term "reducing sugars" is commonly applied indiscriminately to dextrose, levulose, and invert sugar, the same factor being used in calculating either, in mixtures wherein other reducing sugars, as lactose, maltose, etc., having widely different reducing powers are absent.

Fehling's solution is made up in two separate parts as follows:

A. Fehling's Copper Solution.—34.639 grams of carefully selected crystals of pure copper sulphate dissolved in water and diluted to exactly 500 cc.

B. Fehling's Alkaline Tartrate Solution.—178 grams Rochelle salts and 50 grams sodium hydroxide are dissolved in water and diluted to exactly 500 cc.

The Fehling solution should be standardized by dissolving 0.5 gram of pure anhydrous dextrose in water, and diluting to exactly 100 cc. 10 cc. of this dextrose solution should exactly reduce the copper in 10 cc. of the Fehling (5 cc. each of solutions *A* and *B*) when conducted according to the volumetric process described below.

Volumetric Fehling Process.—For determining dextrose, levulose, or invert sugar in a raw or brown sugar, make a solution of the sugar of such a strength that an accurately weighed amount dissolved in water and made up to 100 cc. shall contain not more than 1% of the reducing sugar, as nearly as can be guessed at with reference to the class of sugar under



the solution reaches the dull-red tint, after which care is taken to add a little at a time, keeping account of the total amount added. If the flask be removed from the flame, and the bright, diffused light from a window viewed through the solution with the eye on a level with the surface, a thin film scarcely wider than a line will be observed just below the surface (see Fig. 107), which is blue so long as some of the copper in the solution remains unreduced. When, however, all the copper has been reduced, this film ceases to be blue and becomes colorless or yellow.

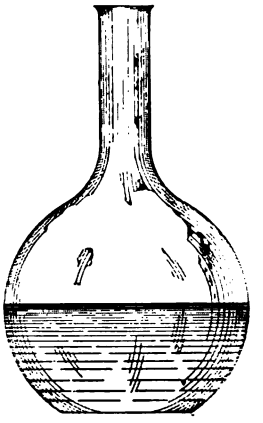


FIG. 107.—Flask and Contents used in Volumetric Fehling Determinations. Showing layer just beneath the surface, the color of which indicates the end-point in adding the sugar-containing liquid.

If the film is not at once apparent, it may often be made quite noticeable by simply diluting the solution in the flask with water. At the approach of the end-point the sugar-containing solution should be added a very little at a time. The exact end-point is best arrived at by decanting a few drops of the mixture in the flask through a filter, acidifying the filtrate with acetic acid, and adding a drop of a solution of ferrocyanide of potassium. As long as there is unreduced copper present, a precipitate or brown-red coloration will appear when the ferrocyanide is added. The sugar solution toward the end should be added to the contents in the flask in small installments (say half a cubic centimeter each time), boiling the liquor for at least three minutes after each addition, until no brown-red coloration is produced by adding the ferrocyanide to a little of the filtered acidified liquid. When the number of cubic centimeters of sugar solution necessary to reduce the copper has thus been determined, a second titration should be made to verify the first, running the entire amount of sugar-containing liquid found necessary in the first case into the second flask.

The equivalents of 10 cc. of Fehling's solution in the above volumetric method are, in terms of the common reducing sugars, as follows:

0.05 gram of { invert sugar, dextrose, or levulose } will reduce 10 cc. Fehling's solution.

0.0475 gram of { cane sugar after inversion } will reduce 10 cc. Fehling's solution.

0.0807 gram of maltose will reduce 10 cc. Fehling's solution.

0.067 gram of lactose " " 10 cc. " "

Suppose, for example, a sample of brown sugar is to be examined for invert sugar. This class of sugar has usually from 2 to 6 per cent of invert sugar. Hence, if 10 grams of the sample are dissolved in 100 cc., the resulting solution will contain not more than 1% of invert sugar.

Suppose 12.9 cc. of this 10% sugar solution were found by the above process to reduce 10 cc. of Fehling's solution.

10 cc. Fehling's solution are equivalent to 0.05 gram invert sugar.

Therefore 12.9 cc. of the sugar solution contain 0.05 gram invert-sugar.

100 cc. sugar solution contain 10 grams sample, and 12.9 cc. contain 1.29 grams sample, the equivalent of 0.05 gram invert sugar.

$$\text{Hence per cent invert sugar} = \frac{0.05 \times 100}{1.29} = 3.9.$$

GRAVIMETRIC FEHLING PROCESSES.—In determining reducing sugars by gravimetric processes, a measured volume of the sugar solution is allowed to act upon a measured volume of hot Fehling's solution for a fixed time, thus forming cuprous oxide. This may be dried and weighed direct, but is more commonly converted either into cupric oxide by ignition, or into metallic copper by reduction with hydrogen or by electrolysis. In any case the sugar is calculated from the weight of the cuprous oxide, the cupric oxide, or the metallic copper (whichever method be used) by the employment of the proper factor, or by the use of tables compiled for the purpose.

Note.—Much difference of opinion exists as to the best and most accurate Fehling gravimetric method to employ. For the determination of dextrose, the Association of Official Agricultural Chemists has given

tables adapted for them all. With the exception of Defren's method and tables, the author knows of no single accurate method adapted without modification to the determination of the various reducing sugars, since in using the tables for dextrose, maltose, and lactose compiled by Allihn, Wein, and Soxhlet, the method employed must in each case be carried out in strict accordance to the minutest details adopted by each of the above authorities, and they are by no means uniform.

The Defren-O'Sullivan Method.*—Mix 15 cc. of Fehling's copper solution, *A* (p. 486), with 15 cc. of the tartrate solution, *B*, in a quarter-liter Erlenmeyer flask, and add 50 cc. of distilled water. Place the flask and its contents in a boiling water bath and allow them to remain five minutes. Then run rapidly from a burette into the hot liquor in the flask 25 cc. of the sugar solution to be tested (which should contain not more than one-half per cent of reducing sugar). Allow the flask to remain in the boiling water bath just fifteen minutes after the addition of the sugar solution, remove, and with the aid of a vacuum filter the contents rapidly in a platinum or porcelain Gooch crucible containing a layer of prepared asbestos fiber about 1 cm. thick, the Gooch with the asbestos having been previously ignited, cooled, and weighed. The cuprous oxide precipitate is thoroughly washed with boiling distilled water till the water ceases to be alkaline.

The asbestos used should be of the long-fibered variety, and should be specially prepared as follows: Boil first with nitric acid (specific gravity 1.05 to 1.10), washing out the acid with hot water, then boil with a 25% solution of sodium hydroxide, and finally wash out the alkali with hot water. Keep the asbestos in a wide-mouthed flask or bottle, and transfer it to the Gooch by shaking it up in the water and pouring it quickly into the crucible while under suction.

Dry the Gooch with its contents in the oven, and finally heat to dull redness for fifteen minutes, during which the red cuprous oxide is converted into the black cupric oxide. If a platinum Gooch is used (and this variety is preferred by the writer), it may be heated directly over the low flame of a burner. If the Gooch is of porcelain, considerable care must be taken to avoid cracking the crucible, the heat being increased cautiously and the operation preferably conducted in a radiator or muffle. After oxidation as above, the crucible is transferred to a desiccator, cooled, and quickly weighed. From the milligrams of cupric oxide, calculate the milligrams of dextrose from the following table:

* Jour. Am. Chem. Soc. 18 (1896), p. 749, and Tech. Quart. 10 (1897), p. 167.

DEFREN'S TABLE FOR THE DETERMINATION OF DEXTROSE, MALTOSE, AND LACTOSE.

Milligrams of Cupric Oxide.	Milligrams of Dextrose.	Milligrams of Maltose.	Milligrams of Lactose.	Milligrams of Cupric Oxide.	Milligrams of Dextrose.	Milligrams of Maltose.	Milligrams of Lactose.
30	13.2	21.7	18.8	80	35.4	58.1	50.5
31	13.7	22.4	19.5	81	35.9	58.9	51.1
32	14.1	23.1	20.1	82	36.3	59.6	51.7
33	14.6	23.9	20.7	83	36.8	60.3	52.4
34	15.0	24.6	21.4	84	37.2	61.1	53.0
35	15.4	25.3	22.0	85	37.7	61.8	53.6
36	15.9	26.1	22.6	86	38.1	62.5	54.3
37	16.3	26.8	23.3	87	38.5	63.3	54.9
38	16.8	27.5	23.9	88	39.0	64.0	55.5
39	17.2	28.3	24.5	89	39.4	64.7	56.2
40	17.6	29.0	25.2	90	39.9	65.5	56.8
41	18.1	29.7	25.8	91	40.3	66.2	57.4
42	18.5	30.5	26.4	92	40.8	66.9	58.1
43	19.0	31.2	27.1	93	41.2	67.7	58.7
44	19.4	31.9	27.7	94	41.7	68.4	59.3
45	19.9	32.7	28.3	95	42.1	69.1	60.0
46	20.3	33.4	29.0	96	42.5	69.9	60.6
47	20.7	34.1	29.6	97	43.0	70.6	61.2
48	21.2	34.8	30.2	98	43.4	71.3	61.9
49	21.6	35.5	30.8	99	43.9	72.1	62.5
50	22.1	36.2	31.5	100	44.4	72.8	63.2
51	22.5	37.0	32.1	101	44.8	73.5	63.8
52	23.0	37.7	32.7	102	45.3	74.3	64.4
53	23.4	38.4	33.3	103	45.7	75.0	65.1
54	23.8	39.2	34.0	104	46.2	75.7	65.7
55	24.2	39.9	34.6	105	46.6	76.5	66.3
56	24.7	40.5	35.2	106	47.0	77.2	67.0
57	25.1	41.3	35.9	107	47.5	77.9	67.6
58	25.5	42.1	36.5	108	48.0	78.7	68.2
59	26.0	42.8	37.1	109	48.4	79.4	68.9
60	26.4	43.5	37.8	110	48.9	80.1	69.5
61	26.9	44.3	38.4	111	49.3	80.9	70.1

DEFREN'S TABLE FOR THE DETERMINATION OF DEXTROSE, MALTOSE, AND LACTOSE—(*Continued*).

Milligrams of Cupric Oxide.	Milligrams of Dextrose.	Milligrams of Maltose.	Milligrams of Lactose.	Milligrams of Cupric Oxide.	Milligrams of Dextrose.	Milligrams of Maltose.	Milligrams of Lactose.
130	57.8	94.8	82.4	180	80.4	131.8	114.6
131	58.2	95.5	83.0	181	80.8	132.5	115.2
132	58.7	96.2	83.6	182	81.3	133.2	115.8
133	59.1	97.0	84.2	183	81.8	134.0	116.5
134	59.6	97.7	84.9	184	82.2	134.7	117.1
135	60.0	98.4	85.5	185	82.7	135.5	117.8
136	60.5	99.2	86.1	186	83.1	136.2	118.4
137	60.9	99.9	86.8	187	83.5	136.9	119.1
138	61.3	100.7	87.4	188	84.0	137.7	119.7
139	61.8	101.4	88.1	189	84.4	138.4	120.4
140	62.2	102.1	88.7	190	84.9	139.1	121.0
141	62.7	102.8	89.3	191	85.4	139.9	121.7
142	63.1	103.5	90.0	192	85.9	140.6	122.3
143	63.6	104.3	90.6	193	86.3	141.4	123.0
144	64.0	105.0	91.3	194	86.8	142.1	123.6
145	64.5	105.8	91.9	195	87.2	142.8	124.3
146	64.9	106.5	92.6	196	87.7	143.6	124.9
147	65.4	107.2	93.2	197	88.1	144.3	125.6
148	65.8	108.0	93.9	198	88.6	145.1	126.2
149	66.3	108.7	94.5	199	89.0	145.8	126.9
150	66.8	109.5	95.2	200	89.5	146.6	127.5
151	67.3	110.2	95.8	201	89.9	147.3	128.2
152	67.7	111.0	96.5	202	90.4	148.1	128.8
153	68.3	111.7	97.1	203	90.8	148.8	129.5
154	68.7	112.4	97.8	204	91.3	149.6	130.1
155	69.2	113.2	98.4	205	91.7	150.3	130.8
156	69.6	113.9	99.1	206	92.2	151.1	131.5
157	70.0	114.7	99.7	207	92.6	151.8	132.1
158	70.5	115.4	100.4	208	93.1	152.5	132.8
159	70.9	116.1	101.0	209	93.5	153.3	133.4
160	71.3	116.9	101.7	210	94.0	154.1	134.1
161	71.8	117.6	102.3	211	94.4	154.8	134.7
162	72.3	118.4	103.0	212	94.9	155.6	135.4
163	72.7	119.1	103.6	213	95.3	156.3	136.0
164	73.2	119.9	104.3	214	95.8	157.1	136.7
165	73.6	120.6	104.9	215	96.3	157.8	137.3
166	74.1	121.4	105.6	216	96.7	158.6	138.0
167	74.5	122.1	106.2	217	97.2	159.3	138.6
168	74.9	122.9	106.9	218	97.6	160.0	139.3
169	75.4	123.6	107.5	219	98.1	160.8	139.9
170	75.8	124.4	108.2	220	98.6	161.5	140.6
171	76.3	125.1	108.8	211	99.0	162.3	141.2
172	76.8	125.8	109.5	222	99.5	163.0	141.9
173	77.3	126.6	110.1	223	99.9	163.7	142.5
174	77.7	127.3	110.8	224	100.4	164.5	143.2
175	78.2	128.1	111.4	225	100.9	165.3	143.8
176	78.6	128.8	112.0	226	101.3	166.0	144.5
177	79.1	129.5	112.6	227	101.8	166.8	145.1
178	79.5	130.3	113.3	228	102.2	167.5	145.8
179	80.0	131.0	113.9	229	102.7	168.3	146.4

DEFREN'S TABLE FOR THE DETERMINATION OF DEXTROSE, MALTOSE, AND LACTOSE—(*Concluded*).

Milligrams of Cupric Oxide.	Milligrams of Dextrose.	Milligrams of Maltose.	Milligrams of Lactose.	Milligrams of Cupric Oxide.	Milligrams of Dextrose.	Milligrams of Maltose.	Milligrams of Lactose.
230	103.1	169.1	147.0	280	126.1	206.8	179.6
231	103.6	169.8	147.7	281	126.5	207.5	180.2
232	104.0	170.6	148.3	282	127.0	208.3	180.9
233	104.5	171.3	149.0	283	127.4	209.0	181.5
234	105.0	172.1	149.6	284	127.9	209.8	182.2
235	105.4	172.8	150.3	285	128.3	210.5	182.9
236	105.9	173.6	150.9	286	128.8	211.3	183.6
237	106.3	174.3	151.6	287	129.3	212.1	184.2
238	106.8	175.1	152.2	288	129.7	212.8	184.9
239	107.2	175.8	152.9	289	130.2	213.6	185.6
240	107.7	176.6	153.5	290	130.6	214.3	186.2
241	108.1	177.3	154.2	291	131.1	215.1	186.9
242	108.6	178.1	154.8	292	131.5	215.9	187.6
243	109.0	178.8	155.5	293	132.0	216.6	188.2
244	109.5	179.6	156.1	294	132.5	217.4	188.9
245	109.9	180.3	156.8	295	133.0	218.2	189.5
246	110.4	181.1	157.4	296	133.4	218.9	190.2
247	110.9	181.8	158.1	297	133.9	219.7	190.8
248	111.3	182.6	158.7	298	134.3	220.4	191.5
249	111.8	183.3	159.4	299	134.8	221.2	192.1
250	112.3	184.1	160.0	300	135.3	221.9	192.8
251	112.7	184.8	160.7	301	135.7	222.7	193.4
252	113.2	185.5	161.3	302	136.2	223.5	194.1
253	113.7	186.3	162.0	303	136.6	224.2	194.7
254	114.1	187.1	162.6	304	137.1	225.0	195.3
255	114.6	187.8	163.3	305	137.6	225.8	196.0
256	115.0	188.6	163.9	306	138.0	226.5	196.6
257	115.5	189.3	164.6	307	138.5	227.3	197.3
258	116.0	190.1	165.2	308	138.9	228.1	197.9
259	116.4	190.8	165.9	309	139.4	228.8	198.6
260	116.9	191.6	166.5	310	139.9	229.6	199.3
261	117.3	192.4	167.2	311	140.3	230.4	199.9



Allihn's Method for the Determination of Dextrose.*—Place 30 cc. of Fehling's copper solution, 30 cc. of the alkaline tartrate solution, and 60 cc. of water in a beaker and heat to boiling. Add 25 cc. of the sugar solution, which must be so prepared as not to contain more than 1% dextrose, and boil over the flame for two minutes. Filter immediately without diluting through a Gooch crucible containing a layer of asbestos fiber, prepared as described on page 489, and wash thoroughly with hot water, using reduced pressure. Transfer the asbestos fiber and the adhering cuprous oxide by means of a glass rod to a beaker and rinse the crucible with about 30 cc. of a boiling mixture of dilute sulphuric and nitric acids containing 65 cc. of sulphuric acid (specific gravity 1.84) and 50 cc. of nitric acid (specific gravity 1.42) per liter. Heat and agitate till the solution is complete, then filter into a scrupulously clean, tared platinum dish of 100 cc. capacity, taking care to wash out all the copper solution from the filter into the dish. Deposit the copper electrolytically in the platinum dish and weigh. Determine the dextrose from Allihn's table, p. 494.

Or, the metallic copper may be calculated by means of the factor 0.7986 from the cupric oxide obtained as in Defren's method (p. 489) and Allihn's table used.

Or, the cuprous oxide as directly obtained by either Allihn's or Defren's method may be washed with alcohol and ether, dried for twenty minutes at 100° C., and weighed, its equivalent in dextrose being ascertained from Allihn's table.

Electrolytic Apparatus.—The author has devised the apparatus shown in Fig. 108 for the electrolytic deposition of copper in sugar analysis and for other work of like nature. *A*, Fig. 109, is a hard-rubber plate 50 cm. long and 25 cm. wide provided with four insulated metal binding posts, *B*, each carrying at the top a thumb screw by which a coiled platinum wire electrode, *C*, may be attached. In front of each post is a copper plate about 4 cm. square covered with thin platinum foil, *P*, which is bent around the edges of the copper plate and so held in place, the copper plate being screwed to the rubber from beneath. On the square platinum-covered plate is set the platinum evaporating-dish which holds the solution from which the copper is to be deposited, the inside of the dish forming the cathode, while the electrode *C*, dipping below the surface of the solution, forms the anode. In front of each platinum-covered plate is a switch, *S*, and at either end of the hard-rubber plate is a binding

* Jour. für praktische Chemie, 22 (1880), p. 46.

ALLIHN'S TABLE FOR THE DETERMINATION OF DEXTROSE.*

Milligrams of Copper.	Milligrams of Cuprous Oxide.	Milligrams of Dextrose.	Milligrams of Copper.	Milligrams of Cuprous Oxide.	Milligrams of Dextrose.	Milligrams of Copper.	Milligrams of Cuprous Oxide.	Milligrams of Dextrose.	Milligrams of Copper.	Milligrams of Cuprous Oxide.	Milligrams of Dextrose.
11	12.4	6.6	76	85.6	38.8	141	158.7	71.8	206	231.9	105.8
12	13.5	7.1	77	86.7	39.3	142	159.9	72.3	207	233.0	106.3
13	14.6	7.6	78	87.8	39.8	143	161.0	72.9	208	234.2	106.8
14	15.8	8.1	79	88.9	40.3	144	162.1	73.4	209	235.3	107.4
15	16.9	8.6	80	90.1	40.8	145	163.2	73.9	210	236.4	107.9
16	18.0	9.0	81	91.2	41.3	146	164.4	74.4	211	237.6	108.4
17	19.1	9.5	82	92.3	41.8	147	165.5	74.9	212	238.7	109.0
18	20.3	10.0	83	93.4	42.3	148	166.6	75.5	213	239.8	109.5
19	21.4	10.5	84	94.6	42.8	149	167.7	76.0	214	240.9	110.0
20	22.5	11.0	85	95.7	43.4	150	168.9	76.5	215	242.1	110.6
21	23.6	11.5	86	96.8	43.9	151	170.0	77.0	216	243.2	111.1
22	24.8	12.0	87	97.9	44.4	152	171.1	77.5	217	244.3	111.6
23	25.9	12.5	88	99.1	44.9	153	172.3	78.1	218	245.4	112.1
24	27.0	13.0	89	100.2	45.4	154	173.4	78.6	219	246.6	112.7
25	28.1	13.5	90	101.3	45.9	155	174.5	79.1	220	247.7	113.2
26	29.3	14.0	91	102.4	46.4	156	175.6	79.6	221	248.7	113.7
27	30.4	14.5	92	103.6	46.9	157	176.8	80.1	222	249.9	114.3
28	31.5	15.0	93	104.7	47.4	158	177.9	80.7	223	251.0	114.8
29	32.7	15.5	94	105.8	47.9	159	179.0	81.2	224	252.4	115.3
30	33.8	16.0	95	107.0	48.4	160	180.1	81.7	225	253.3	115.9
31	34.9	16.5	96	108.1	48.9	161	181.3	82.2	226	254.4	116.4
32	36.0	17.0	97	109.2	49.4	162	182.4	82.7	227	255.6	116.9
33	37.2	17.5	98	110.3	49.9	163	183.5	83.3	228	256.7	117.4
34	38.3	18.0	99	111.5	50.4	164	184.6	83.8	229	257.8	118.0
35	39.4	18.5	100	112.6	50.9	165	185.8	84.3	230	258.9	118.5
36	40.5	18.9	101	113.7	51.4	166	186.9	84.8	231	260.1	119.0
37	41.7	19.4	102	114.8	51.9	167	188.0	85.3	232	261.2	119.6
38	42.8	19.9	103	116.0	52.4	168	189.1	85.9	233	262.3	120.1
39	43.9	20.4	104	117.1	52.9	169	190.3	86.4	234	263.4	120.7
40	45.0	20.9	105	118.2	53.5	170	191.4	86.9	235	264.6	121.2
41	46.2	21.4	106	119.3	54.0	171	192.5	87.4	236	265.7	121.7
42	47.3	21.9	107	120.5	54.5	172	193.6	87.9	237	266.8	122.3
43	48.4	22.4	108	121.6	55.0	173	194.8	88.5	238	268.0	122.8
44	49.5	22.9	109	122.7	55.5	174	195.9	89.0	239	269.1	123.4
45	50.7	23.4	110	123.8	56.0	175	197.0	89.5	240	270.2	123.9
46	51.8	23.9	111	125.0	56.5	176	198.1	90.0	241	271.3	124.4
47	52.9	24.4	112	126.1	57.0	177	199.3	90.5	242	272.5	125.0
48	54.0	24.9	113	127.2	57.5	178	200.4	91.1	243	273.6	125.5
49	55.2	25.4	114	128.3	58.0	179	201.5	91.6	244	274.7	126.0
50	56.3	25.9	115	129.6	58.6	180	202.6	92.1	245	275.8	126.6
51	57.4	26.4	116	130.6	59.1	181	203.8	92.6	246	277.0	127.1
52	58.5	26.9	117	131.7	59.6	182	204.9	93.1	247	278.1	127.6



ALLIHN'S TABLE FOR THE DETERMINATION OF DEXTROSE—(*Continued*).

Milligrams of Copper.	Milligrams of Cuprous Oxide.	Milligrams of Dextrose.	Milligrams of Copper.	Milligrams of Cuprous Oxide.	Milligrams of Dextrose.	Milligrams of Copper.	Milligrams of Cuprous Oxide.	Milligrams of Dextrose.	Milligrams of Copper.	Milligrams of Cuprous Oxide.	Milligrams of Dextrose.
271	305.1	140.6	321	361.4	168.1	371	417.7	196.3	421	474.0	225.1
272	306.2	141.1	322	362.5	168.6	372	418.8	196.8	422	475.6	225.7
273	307.3	141.7	323	363.7	169.2	373	420.0	197.4	423	476.2	226.3
274	308.5	142.2	324	364.8	169.7	374	421.1	198.0	424	477.4	226.9
275	309.6	142.8	325	365.9	170.3	375	422.2	198.6	425	478.5	227.5
276	310.7	143.3	326	367.0	170.9	376	423.3	199.1	426	479.6	228.0
277	311.9	143.9	327	368.2	171.4	377	424.5	199.7	427	480.7	228.6
278	313.0	144.4	328	369.3	172.0	378	425.6	200.3	428	481.9	229.2
279	314.1	145.0	329	370.4	172.5	379	426.7	200.8	429	483.0	229.8
280	315.2	145.5	330	371.5	173.1	380	427.8	201.4	430	484.1	230.4
281	316.4	146.1	331	372.7	173.7	381	429.0	202.0	431	485.3	231.0
282	317.5	146.6	332	373.8	174.2	382	430.1	202.5	432	486.4	231.6
283	318.6	147.2	333	374.9	174.8	383	431.2	203.1	433	487.5	232.2
284	319.7	147.7	334	376.0	175.3	384	432.3	203.7	434	488.6	232.8
285	320.9	148.3	335	377.2	175.9	385	433.5	204.3	435	489.7	233.4
286	322.0	148.8	336	378.3	176.5	386	434.6	204.8	436	490.9	233.9
287	323.1	149.4	337	379.4	177.0	387	435.7	205.4	437	492.0	234.5
288	324.2	149.9	338	380.5	177.6	388	436.8	206.0	438	493.1	235.1
289	325.4	150.5	339	381.7	178.1	389	438.0	206.5	439	494.3	235.7
290	326.5	151.0	340	382.8	178.7	390	439.1	207.1	440	495.4	236.3
291	327.4	151.6	341	383.9	179.3	391	440.2	207.7	441	496.5	236.9
292	328.7	152.1	342	385.0	179.8	392	441.3	208.3	442	497.6	237.5
293	329.9	152.7	343	386.2	180.4	393	442.4	208.8	443	498.8	238.1
294	331.0	153.2	344	387.3	180.9	394	443.6	209.4	444	499.9	238.7
295	332.1	153.8	345	388.4	181.5	395	444.7	210.0	445	501.0	239.3
296	333.3	154.3	346	389.6	182.1	396	445.9	210.6	446	502.1	239.8
297	334.4	154.9	347	390.7	182.6	397	447.0	211.2	447	503.2	240.4
298	335.5	155.4	348	391.8	183.2	398	448.1	211.7	448	504.4	241.0
299	336.6	156.0	349	392.9	183.7	399	449.2	212.3	449	505.5	241.6
300	337.8	156.5	350	394.0	184.3	400	450.3	212.9	450	506.6	242.2
301	338.9	157.1	351	395.2	184.9	401	451.5	213.5	451	507.8	242.8
302	340.0	157.6	352	396.3	185.4	402	452.6	214.1	452	508.9	243.4
303	341.1	158.2	353	397.4	186.0	403	453.7	214.6	453	510.0	244.0
304	342.3	158.7	354	398.6	186.6	404	454.8	215.2	454	511.1	244.6
305	343.4	159.3	355	399.7	187.2	405	456.0	215.8	455	512.3	245.2
306	344.5	159.8	356	400.8	187.7	406	457.1	216.4	456	513.4	245.7
307	345.6	160.4	357	401.9	188.3	407	458.2	217.0	457	514.5	246.3
308	346.8	160.9	358	403.1	188.9	408	459.4	217.5	458	515.6	246.9
309	347.9	161.5	359	404.2	189.4	409	460.5	218.1	459	516.8	247.5
310	349.0	162.0	360	405.3	190.0	410	461.6	218.7	460	517.9	248.1
311	350.1	162.6	361	406.4	190.6	411	462.7	219.3	461	519.0	248.7
312	351.3	163.1	362	407.6	191.1	412	463.8	219.9	462	520.1	249.3
313	352.4	163.7	363	408.7	191.7	413	465.0	220.4	463	521.3	249.9
314	353.5	164.2	364	409.8	192.3	414	466.1	221.0			
315	354.6	164.8	365	410.9	192.9	415	467.2	221.6			
316	355.8	165.3	366	412.1	193.4	416	468.4	222.2			
317	356.9	165.9	367	413.2	194.0	417	469.5	222.8			
318	358.0	166.4	368	414.3	194.6	418	470.6	223.3			
319	359.1	167.0	369	415.4	195.1	419	471.8	223.9			
320	360.3	167.5	370	416.6	195.7	420	472.9	224.5			

post, *R*, for connection with the electric current. The wiring, which is on the under side of the rubber plate, is best illustrated by the diagram in Fig. 109.

Four determinations may be carried on simultaneously in four platinum dishes, if desired, the wiring and the switches being so arranged that beginning at one end of the plate either the first dish or the first

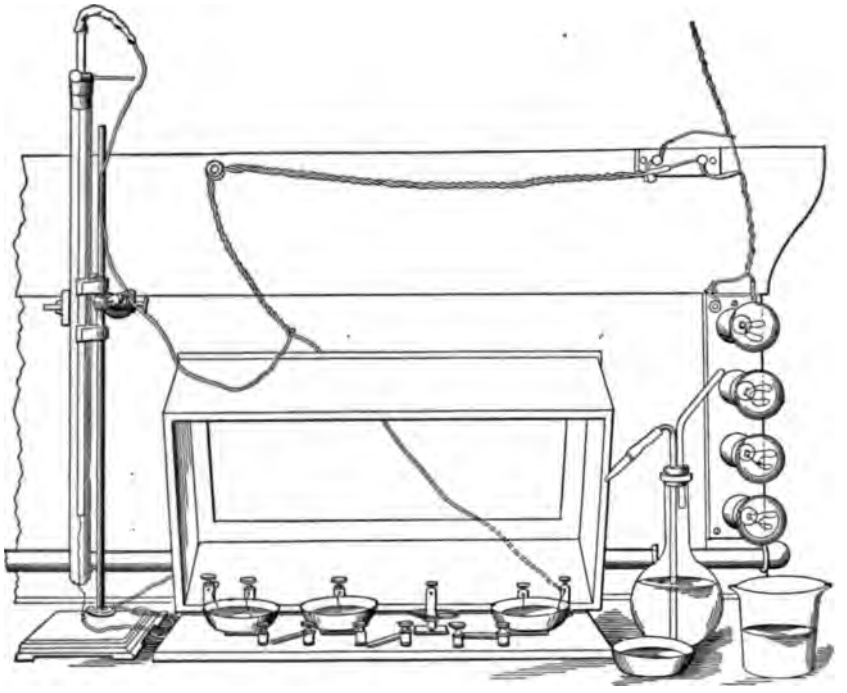
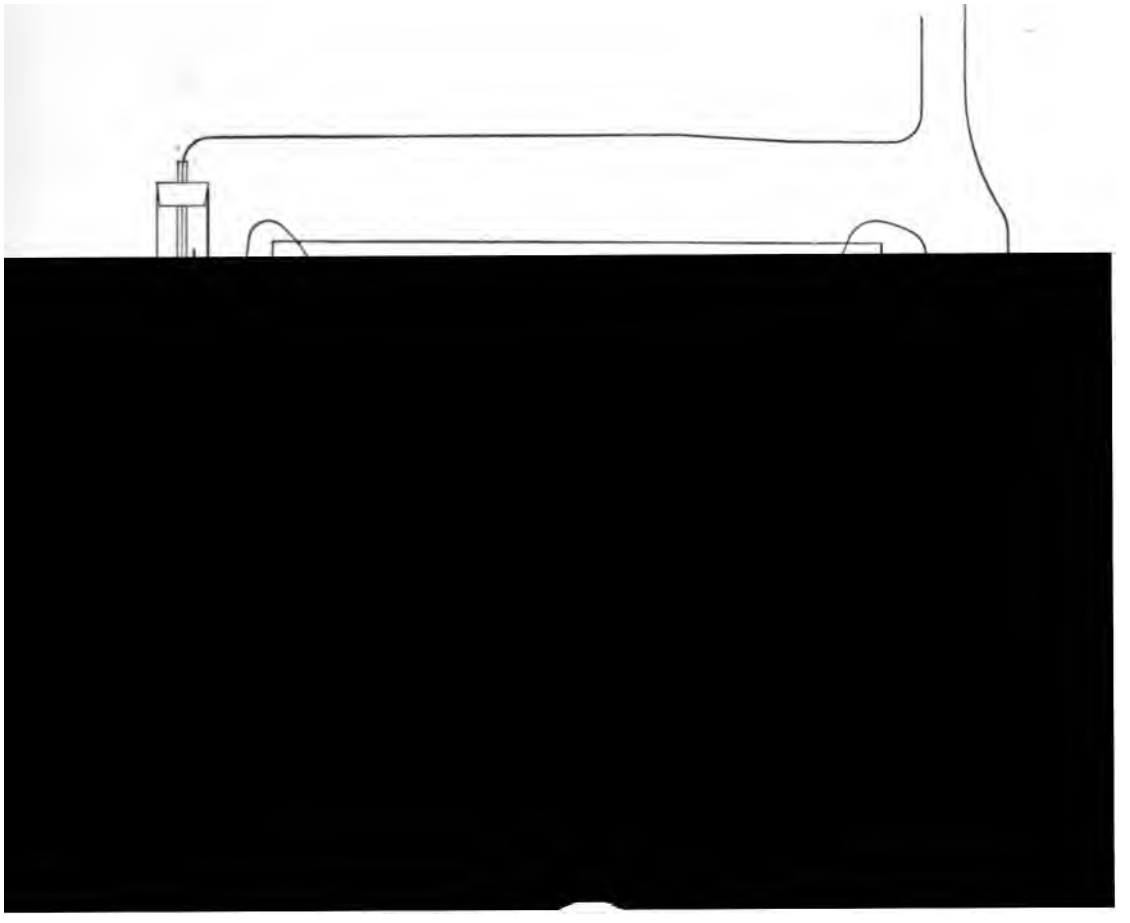


FIG. 108.—Electrolytic Apparatus, with Glass-covered Top Partially Removed.



two or the first three may be thrown in or out of circuit at will without interrupting the current through the remaining dishes. A cover with wooden sides and glass top fits closely over the whole apparatus as a protection from dust, but may be easily lifted off to manipulate the dishes when desired. The sides of the cover are perforated to permit the escape of the gas formed during the electrolysis.

The ordinary street current is used when available, and the strength of the current may be varied within wide limits by means of a number of 16 or 32 candle-power lamps, *K*, coupled in multiple, and a rheostat, *L*, consisting of a vertical glass tube sealed at the bottom, containing a column of dilute acid, the resistance being changed by varying the length of the acid column contained between the two platinum terminals immersed therein, one of which is movable. A gravity battery of four cells may be employed if the laboratory is not equipped with electric lights.

In using this apparatus for determining copper, as in sugar work, the plating process should go on till all the copper is deposited, requiring several hours or over night with a current strength of about 0.25 ampere. Before stopping the process, the absence of copper in the solution should be proved by removing a few drops with a pipette, adding first ammonia, then acetic acid, and testing with ferrocyanide of potassium. If no brown coloration is produced, all the copper has been plated out. Throw the dish out of circuit by means of the switch, pour out the acid solution quickly before it has a chance to dissolve any of the copper, wash the dish first with water and then with alcohol, dry, and weigh.

The copper may be removed from the platinum dish by strong nitric acid.

Determination of Sucrose by Fehling's Solution.*—If a polariscope is not available, cane sugar can be determined as follows: First determine the percentage of invert sugar present in the sample by one of the Fehling methods already described. Then dissolve 1 gram of the sugar in about 100 cc. of water in a 500-cc. graduated flask, add 3 cc. of concentrated hydrochloric acid and invert by heating in water to 68° and cooling in the regular manner. Neutralize with sodium hydroxide or sodium carbonate, and make up to the mark with water. Determine the per cent of total reducing sugar as invert sugar either by the volumetric or gravimetric Fehling process. Subtract the invert sugar found present in the sugar by direct determination from the total found present after inversion, and

* Tucker, Manual of Sugar Analysis, p. 182.

the remainder is the invert sugar due to cane sugar. This figure multiplied by 0.95 gives the percentage of cane sugar.

For the determination of sucrose by the gravimetric Fehling process on the inverted sample, multiply the cupric oxide (CuO) by the factor 0.4307, or the copper (Cu) by the factor 0.5394.

Test for Ultramarine in Sugar.*—A large amount of the sugar is dissolved in water and the coloring matter is allowed to settle out, washing the residue several times by decantation. On treatment with hydrochloric acid, the blue color is discharged if due to ultramarine.

Analysis of Molasses and Syrups.—First insure a perfectly homogeneous sample by stirring with a rod to evenly distribute any separated sugar.

Determination of Total Solids. — (1) *Direct Method.* — Weigh 20 grams into a 100-cc. graduated flask, dissolve in water, and make up to the mark. Insure a uniform solution by shaking. Measure 10 cc. of this solution into a tared platinum dish containing about 5 grams of freshly ignited, finely divided asbestos fiber, and dry to constant weight at 70° *in vacuo*, or in a McGill oven (see p. 481).

(2) *By Calculation from Specific Gravity.*—Weigh 25 grams of the sample into a 100-cc. graduated flask, dissolve in water, and make up to the mark. Determine the specific gravity of the diluted solution by means of a pycnometer or Westphal balance. Ascertain from the accompanying table the percentage by weight of solids (sugar) corresponding to the specific gravity of the diluted solution, and calculate the total solids in the original sample by the formula

$$\text{Solids in original sample} = 4DS,$$



RELATION OF BRIX, SPECIFIC GRAVITY, AND BAUMÉ.

Per Cent of Sugar.	Specific Gravity.	Degree Baumé.	Per Cent of Sugar.	Specific Gravity.	Degree Baumé.	Per Cent of Sugar.	Specific Gravity.	Degree Baumé.	Per Cent of Sugar.	Specific Gravity.	Degree Baumé.
0.1	1.0003	0.06	6.6	1.0261	3.7	13.1	1.0531	7.3	19.6	1.0815	10.85
0.2	1.0007	0.11	6.7	1.0265	3.7	13.2	1.0536	7.3	19.7	1.0819	10.9
0.3	1.0011	0.17	6.8	1.0269	3.8	13.3	1.0540	7.4	19.8	1.0824	11.0
0.4	1.0015	0.22	6.9	1.0273	3.8	13.4	1.0544	7.4	19.9	1.0828	11.0
0.5	1.0019	0.28	7.0	1.0277	3.9	13.5	1.0548	7.5	20.0	1.0832	11.1
0.6	1.0023	0.33	7.1	1.0281	3.9	13.6	1.0553	7.5	20.1	1.0837	11.1
0.7	1.0027	0.39	7.2	1.0286	4.0	13.7	1.0557	7.6	20.2	1.0841	11.2
0.8	1.0031	0.44	7.3	1.0290	4.1	13.8	1.0561	7.65	20.3	1.0846	11.2
0.9	1.0034	0.5	7.4	1.0294	4.1	13.9	1.0566	7.7	20.4	1.0850	11.3
1.0	1.0038	0.55	7.5	1.0298	4.2	14.0	1.0570	7.8	20.5	1.0855	11.3
1.1	1.0042	0.6	7.6	1.0302	4.2	14.1	1.0574	7.8	20.6	1.0859	11.4
1.2	1.0046	0.7	7.7	1.0306	4.3	14.2	1.0578	7.9	20.7	1.0864	11.45
1.3	1.0050	0.7	7.8	1.0310	4.3	14.3	1.0583	7.9	20.8	1.0868	11.5
1.4	1.0054	0.8	7.9	1.0314	4.4	14.4	1.0587	8.0	20.9	1.0873	11.6
1.5	1.0058	0.8	8.0	1.0318	4.4	14.5	1.0591	8.0	21.0	1.0877	11.6
1.6	1.0062	0.9	8.1	1.0322	4.5	14.6	1.0596	8.1	21.1	1.0882	11.7
1.7	1.0066	0.9	8.2	1.0327	4.55	14.7	1.0600	8.15	21.2	1.0886	11.7
1.8	1.0070	1.0	8.3	1.0331	4.6	14.8	1.0604	8.2	21.3	1.0891	11.8
1.9	1.0074	1.05	8.4	1.0335	4.7	14.9	1.0609	8.3	21.4	1.0895	11.8
2.0	1.0077	1.1	8.5	1.0339	4.7	15.0	1.0613	8.3	21.5	1.0900	11.9
2.1	1.0081	1.2	8.6	1.0343	4.8	15.1	1.0617	8.4	21.6	1.0904	11.95
2.2	1.0085	1.2	8.7	1.0347	4.8	15.2	1.0621	8.4	21.7	1.0909	12.0
2.3	1.0089	1.3	8.8	1.0351	4.9	15.3	1.0626	8.5	21.8	1.0914	12.05
2.4	1.0093	1.3	8.9	1.0355	4.9	15.4	1.0630	8.5	21.9	1.0918	12.1
2.5	1.0097	1.4	9.0	1.0359	5.0	15.5	1.0634	8.6	22.0	1.0923	12.2
2.6	1.0101	1.4	9.1	1.0364	5.05	15.6	1.0639	8.65	22.1	1.0927	12.2
2.7	1.0105	1.5	9.2	1.0368	5.1	15.7	1.0643	8.7	22.2	1.0932	12.3
2.8	1.0109	1.55	9.3	1.0372	5.2	15.8	1.0647	8.8	22.3	1.0936	12.3
2.9	1.0113	1.6	9.4	1.0376	5.2	15.9	1.0652	8.8	22.4	1.0941	12.4
3.0	1.0117	1.7	9.5	1.0380	5.3	16.0	1.0656	8.9	22.5	1.0945	12.4
3.1	1.0121	1.7	9.6	1.0384	5.3	16.1	1.0660	8.9	22.6	1.0950	12.5
3.2	1.0125	1.8	9.7	1.0388	5.4	16.2	1.0665	9.0	22.7	1.0954	12.55
3.3	1.0129	1.8	9.8	1.0393	5.4	16.3	1.0669	9.0	22.8	1.0959	12.6
3.4	1.0133	1.9	9.9	1.0397	5.5	16.4	1.0674	9.1	22.9	1.0964	12.7
3.5	1.0137	1.9	10.0	1.0401	5.55	16.5	1.0678	9.1	23.0	1.0968	12.7
3.6	1.0141	2.0	10.1	1.0405	5.6	16.6	1.0682	9.2	23.1	1.0973	12.8
3.7	1.0145	2.0	10.2	1.0409	5.7	16.7	1.0687	9.25	23.2	1.0977	12.8
3.8	1.0149	2.1	10.3	1.0413	5.7	16.8	1.0691	9.3	23.3	1.0982	12.9
3.9	1.0153	2.2	10.4	1.0418	5.8	16.9	1.0695	9.4	23.4	1.0986	12.9
4.0	1.0157	2.2	10.5	1.0422	5.8	17.0	1.0700	9.4	23.5	1.0991	13.0
4.1	1.0161	2.3	10.6	1.0426	5.9	17.1	1.0704	9.5	23.6	1.0996	13.0
4.2	1.0165	2.3	10.7	1.0430	5.9	17.2	1.0709	9.5	23.7	1.1000	13.1
4.3	1.0169	2.4	10.8	1.0434	6.0	17.3	1.0713	9.6	23.8	1.1005	13.15
4.4	1.0173	2.4	10.9	1.0439	6.05	17.4	1.0717	9.6	23.9	1.1009	13.2
4.5	1.0177	2.5	11.0	1.0443	6.1	17.5	1.0722	9.7	24.0	1.1014	13.3
4.6	1.0181	2.6	11.1	1.0447	6.2	17.6	1.0726	9.75	24.1	1.1019	13.3
4.7	1.0185	2.6	11.2	1.0451	6.2	17.7	1.0730	9.8	24.2	1.1023	13.4
4.8	1.0189	2.7	11.3	1.0455	6.3	17.8	1.0735	9.9	24.3	1.1028	13.4
4.9	1.0193	2.7	11.4	1.0459	6.3	17.9	1.0739	9.9	24.4	1.1032	13.5
5.0	1.0197	2.8	11.5	1.0464	6.4	18.0	1.0744	10.0	24.5	1.1037	13.5
5.1	1.0201	2.8	11.6	1.0468	6.4	18.1	1.0748	10.0	24.6	1.1042	13.6
5.2	1.0205	2.9	11.7	1.0472	6.5	18.2	1.0753	10.1	24.7	1.1046	13.6
5.3	1.0209	2.9	11.8	1.0476	6.55	18.3	1.0757	10.1	24.8	1.1051	13.7
5.4	1.0213	3.0	11.9	1.0481	6.6	18.4	1.0761	10.2	24.9	1.1056	13.75
5.5	1.0217	3.0	12.0	1.0485	6.7	18.5	1.0766	10.2	25.0	1.1060	13.8
5.6	1.0221	3.1	12.1	1.0489	6.7	18.6	1.0770	10.3	25.1	1.1065	13.9
5.7	1.0225	3.2	12.2	1.0493	6.8	18.7	1.0775	10.35	25.2	1.1070	13.9
5.8	1.0229	3.2	12.3	1.0497	6.8	18.8	1.0779	10.4	25.3	1.1074	14.0
5.9	1.0233	3.3	12.4	1.0502	6.9	18.9	1.0783	10.5	25.4	1.1079	14.0
6.0	1.0237	3.3	12.5	1.0506	6.9	19.0	1.0788	10.5	25.5	1.1083	14.1
6.1	1.0241	3.4	12.6	1.0510	7.0	19.1	1.0792	10.6	25.6	1.1088	14.1
6.2	1.0245	3.4	12.7	1.0514	7.05	19.2	1.0797	10.6	25.7	1.1093	14.2
6.3	1.0249	3.5	12.8	1.0519	7.1	19.3	1.0801	10.7	25.8	1.1097	14.2
6.4	1.0253	3.6	12.9	1.0523	7.2	19.4	1.0806	10.7	25.9	1.1102	14.3
6.5	1.0257	3.6	13.0	1.0527	7.2	19.5	1.0810	10.8	26.0	1.1107	14.35

RELATION OF BRIX, SPECIFIC GRAVITY, AND BAUMÉ—(*Continued*).

Per Cent of Sugar.	Specific Gravity.	Degree Baumé.	Per Cent of Sugar.	Specific Gravity.	Degree Baumé.	Per Cent of Sugar.	Specific Gravity.	Degree Baumé.	Per Cent of Sugar.	Specific Gravity.	Degree Baumé.
26.1	1.1111	14.4	32.6	1.1422	17.9	39.1	1.1748	21.4	45.6	1.2088	24.9
26.2	1.1116	14.5	32.7	1.1427	18.0	39.2	1.1753	21.5	45.7	1.2093	24.9
26.3	1.1121	14.5	32.8	1.1432	18.0	39.3	1.1758	21.5	45.8	1.2099	25.0
26.4	1.1125	14.6	32.9	1.1437	18.1	39.4	1.1763	21.6	45.9	1.2104	25.0
26.5	1.1130	14.6	33.0	1.1442	18.15	39.5	1.1768	21.6	46.0	1.2110	25.1
26.6	1.1135	14.7	33.1	1.1447	18.2	39.6	1.1773	21.7	46.1	1.2115	25.1
26.7	1.1140	14.7	33.2	1.1452	18.25	39.7	1.1778	21.7	46.2	1.2120	25.2
26.8	1.1144	14.8	33.3	1.1457	18.3	39.8	1.1784	21.8	46.3	1.2126	25.2
26.9	1.1140	14.8	33.4	1.1462	18.4	39.9	1.1789	21.85	46.4	1.2131	25.3
27.0	1.1154	14.9	33.5	1.1466	18.4	40.0	1.1794	21.9	46.5	1.2136	25.35
27.1	1.1158	14.9	33.6	1.1471	18.5	40.1	1.1799	22.0	46.6	1.2142	25.4
27.2	1.1163	15.0	33.7	1.1476	18.5	40.2	1.1804	22.0	46.7	1.2147	25.45
27.3	1.1168	15.1	33.8	1.1481	18.6	40.3	1.1809	22.1	46.8	1.2153	25.5
27.4	1.1172	15.1	33.9	1.1486	18.6	40.4	1.1815	22.1	46.9	1.2158	25.6
27.5	1.1177	15.2	34.0	1.1491	18.7	40.5	1.1820	22.2	47.0	1.2163	25.6
27.6	1.1182	15.2	34.1	1.1496	18.7	40.6	1.1825	22.2	47.1	1.2169	25.7
27.7	1.1187	15.3	34.2	1.1501	18.8	40.7	1.1830	22.3	47.2	1.2174	25.7
27.8	1.1191	15.3	34.3	1.1506	18.85	40.8	1.1835	22.3	47.3	1.2180	25.8
27.9	1.1196	15.4	34.4	1.1511	18.9	40.9	1.1840	22.4	47.4	1.2185	25.8
28.0	1.1201	15.4	34.5	1.1516	18.95	41.0	1.1846	22.4	47.5	1.2191	25.9
28.1	1.1206	15.5	34.6	1.1521	19.0	41.1	1.1851	22.5	47.6	1.2196	25.9
28.2	1.1210	15.55	34.7	1.1526	19.1	41.2	1.1856	22.5	47.7	1.2201	26.0
28.3	1.1215	15.6	34.8	1.1531	19.1	41.3	1.1861	22.6	47.8	1.2207	26.0
28.4	1.1220	15.7	34.9	1.1536	19.2	41.4	1.1866	22.65	47.9	1.2212	26.1
28.5	1.1225	15.7	35.0	1.1541	19.2	41.5	1.1872	22.7	48.0	1.2218	26.1
28.6	1.1229	15.8	35.1	1.1546	19.3	41.6	1.1877	22.75	48.1	1.2223	26.2
28.7	1.1234	15.8	35.2	1.1551	19.3	41.7	1.1882	22.8	48.2	1.2229	26.2
28.8	1.1230	15.9	35.3	1.1556	19.4	41.8	1.1887	22.9	48.3	1.2234	26.3
28.9	1.1244	15.9	35.4	1.1561	19.4	41.9	1.1892	22.9	48.4	1.2240	26.35
29.0	1.1248	16.0	35.5	1.1566	19.5	42.0	1.1898	23.0	48.5	1.2245	26.4
29.1	1.1253	16.0	35.6	1.1571	19.55	42.1	1.1903	23.0	48.6	1.2250	26.45
29.2	1.1258	16.1	35.7	1.1576	19.6	42.2	1.1908	23.1	48.7	1.2256	26.5
29.3	1.1263	16.1	35.8	1.1581	19.65	42.3	1.1913	23.1	48.8	1.2261	26.6
29.4	1.1267	16.2	35.9	1.1586	19.7	42.4	1.1919	23.2	48.9	1.2267	26.6
29.5	1.1272	16.25	36.0	1.1591	19.8	42.5	1.1924	23.2	49.0	1.2272	26.7
29.6	1.1277	16.3	36.1	1.1596	19.8	42.6	1.1929	23.3	49.1	1.2278	26.7
29.7	1.1282	16.4	36.2	1.1601	19.9	42.7	1.1934	23.3	49.2	1.2283	26.8
29.8	1.1287	16.4	36.3	1.1606	19.9	42.8	1.1940	23.4	49.3	1.2289	26.8
29.9	1.1291	16.5	36.4	1.1611	20.0	42.9	1.1945	23.45	49.4	1.2294	26.9
30.0	1.1296	16.5	36.5	1.1616	20.0	43.0	1.1950	23.5	49.5	1.2300	26.9
30.1	1.1301	16.6	36.6	1.1621	20.1	43.1	1.1955	23.55	49.6	1.2305	27.0
30.2	1.1306	16.6	36.7	1.1626	20.1	43.2	1.1961	23.6	49.7	1.2311	27.0
30.3	1.1311	16.7	36.8	1.1631	20.2	43.3	1.1966	23.7	49.8	1.2316	27.1

RELATION OF BRIX, SPECIFIC GRAVITY, AND BAUMÉ—(*Continued*).

Per Cent of Sugar.	Specific Gravity.	Degree Baumé.	Per Cent of Sugar.	Specific Gravity.	Degree Baumé.	Per Cent of Sugar.	Specific Gravity.	Degree Baumé.	Per Cent of Sugar.	Specific Gravity.	Degree Baumé.
52.1	1.2444	28.3	58.6	1.2816	31.6	65.1	1.3205	34.05	71.6	1.3610	38.2
52.2	1.2450	28.3	58.7	1.2822	31.7	65.2	1.3211	35.0	71.7	1.3615	38.2
52.3	1.2455	28.4	58.8	1.2828	31.7	65.3	1.3217	35.05	71.8	1.3623	38.2
52.4	1.2461	28.4	58.9	1.2834	31.8	65.4	1.3223	35.1	71.9	1.3629	38.3
52.5	1.2467	28.5	59.0	1.2840	31.85	65.5	1.3229	35.15	72.0	1.3635	38.3
52.6	1.2472	28.5	59.1	1.2845	31.9	65.6	1.3235	35.2	72.1	1.3642	38.4
52.7	1.2478	28.6	59.2	1.2851	31.95	65.7	1.3241	35.25	72.2	1.3648	38.4
52.8	1.2483	28.65	59.3	1.2857	32.0	65.8	1.3247	35.3	72.3	1.3655	38.5
52.9	1.2489	28.7	59.4	1.2863	32.05	65.9	1.3253	35.35	72.4	1.3661	38.5
53.0	1.2495	28.75	59.5	1.2869	32.1	66.0	1.3260	35.4	72.5	1.3667	38.6
53.1	1.2500	28.8	59.6	1.2875	32.15	66.1	1.3266	35.4	72.6	1.3674	38.6
53.2	1.2506	28.85	59.7	1.2881	32.2	66.2	1.3272	35.5	72.7	1.3680	38.7
53.3	1.2512	28.9	59.8	1.2887	32.3	66.3	1.3278	35.5	72.8	1.3687	38.7
53.4	1.2517	28.9	59.9	1.2893	32.3	66.4	1.3285	35.6	72.9	1.3693	38.8
53.5	1.2523	29.0	60.0	1.2898	32.4	66.5	1.3291	35.6	73.0	1.3699	38.8
53.6	1.2529	29.1	60.1	1.2904	32.4	66.6	1.3297	35.7	73.1	1.3705	38.9
53.7	1.2534	29.1	60.2	1.2910	32.5	66.7	1.3303	35.7	73.2	1.3712	38.9
53.8	1.2540	29.2	60.3	1.2916	32.5	66.8	1.3309	35.8	73.3	1.3719	39.0
53.9	1.2546	29.2	60.4	1.2922	32.6	66.9	1.3315	35.8	73.4	1.3725	39.0
54.0	1.2551	29.3	60.5	1.2928	32.6	67.0	1.3322	35.9	73.5	1.3732	39.1
54.1	1.2557	29.3	60.6	1.2934	32.7	67.1	1.3327	35.9	73.6	1.3738	39.1
54.2	1.2563	29.4	60.7	1.2940	32.7	67.2	1.3334	36.0	73.7	1.3745	39.2
54.3	1.2568	29.4	60.8	1.2946	32.8	67.3	1.3340	36.0	73.8	1.3751	39.2
54.4	1.2574	29.5	60.9	1.2952	32.8	67.4	1.3346	36.1	73.9	1.3757	39.3
54.5	1.2580	29.5	61.0	1.2958	32.9	67.5	1.3352	36.1	74.0	1.3764	39.3
54.6	1.2585	29.6	61.1	1.2964	32.9	67.6	1.3359	36.2	74.1	1.3770	39.4
54.7	1.2591	29.6	61.2	1.2970	33.0	67.7	1.3365	36.2	74.2	1.3777	39.4
54.8	1.2597	29.7	61.3	1.2975	33.0	67.8	1.3371	36.3	74.3	1.3783	39.5
54.9	1.2602	29.7	61.4	1.2981	33.1	67.9	1.3377	36.3	74.4	1.3790	39.5
55.0	1.2608	29.8	61.5	1.2987	33.1	68.0	1.3384	36.4	74.5	1.3796	39.6
55.1	1.2614	29.8	61.6	1.2993	33.2	68.1	1.3390	36.4	74.6	1.3803	39.6
55.2	1.2620	29.9	61.7	1.2999	33.2	68.2	1.3396	36.5	74.7	1.3809	39.7
55.3	1.2625	29.9	61.8	1.3005	33.3	68.3	1.3402	36.5	74.8	1.3816	39.7
55.4	1.2631	30.0	61.9	1.3011	33.3	68.4	1.3408	36.6	74.9	1.3822	39.8
55.5	1.2637	30.05	62.0	1.3017	33.4	68.5	1.3415	36.6	75.0	1.3828	39.8
55.6	1.2642	30.1	62.1	1.3023	33.4	68.6	1.3421	36.7	75.1	1.3835	39.9
55.7	1.2648	30.15	62.2	1.3029	33.5	68.7	1.3427	36.7	75.2	1.3842	39.9
55.8	1.2654	30.2	62.3	1.3035	33.5	68.8	1.3433	36.8	75.3	1.3848	40.0
55.9	1.2660	30.25	62.4	1.3040	33.6	68.9	1.3440	36.8	75.4	1.3855	40.0
56.0	1.2665	30.3	62.5	1.3047	33.6	69.0	1.3446	36.9	75.5	1.3861	40.1
56.1	1.2671	30.4	62.6	1.3053	33.7	69.1	1.3452	36.9	75.6	1.3868	40.1
56.2	1.2677	30.4	62.7	1.3059	33.7	69.2	1.3458	37.0	75.7	1.3874	40.2
56.3	1.2683	30.5	62.8	1.3065	33.8	69.3	1.3465	37.0	75.8	1.3881	40.2
56.4	1.2688	30.5	62.9	1.3071	33.8	69.4	1.3471	37.1	75.9	1.3887	40.3
56.5	1.2694	30.6	63.0	1.3077	33.9	69.5	1.3477	37.1	76.0	1.3894	40.3
56.6	1.2700	30.6	63.1	1.3083	33.9	69.6	1.3484	37.2	76.1	1.3900	40.4
56.7	1.2706	30.7	63.2	1.3089	34.0	69.7	1.3490	37.2	76.2	1.3907	40.4
56.8	1.2712	30.7	63.3	1.3095	34.0	69.8	1.3496	37.3	76.3	1.3913	40.5
56.9	1.2717	30.8	63.4	1.3101	34.1	69.9	1.3502	37.3	76.4	1.3920	40.5
57.0	1.2723	30.8	63.5	1.3107	34.1	70.0	1.3509	37.4	76.5	1.3926	40.6
57.1	1.2729	30.9	63.6	1.3113	34.2	70.1	1.3515	37.4	76.6	1.3933	40.6
57.2	1.2735	30.9	63.7	1.3119	34.2	70.2	1.3521	37.5	76.7	1.3939	40.7
57.3	1.2741	31.0	63.8	1.3125	34.3	70.3	1.3528	37.5	76.8	1.3946	40.7
57.4	1.2747	31.0	63.9	1.3132	34.3	70.4	1.3534	37.6	76.9	1.3953	40.8
57.5	1.2752	31.1	64.0	1.3138	34.4	70.5	1.3540	37.6	77.0	1.3959	40.8
57.6	1.2758	31.1	64.1	1.3144	34.4	70.6	1.3546	37.7	77.1	1.3966	40.8
57.7	1.2764	31.2	64.2	1.3150	34.5	70.7	1.3553	37.7	77.2	1.3972	40.9
57.8	1.2769	31.2	64.3	1.3156	34.5	70.8	1.3559	37.8	77.3	1.3979	41.0
57.9	1.2775	31.3	64.4	1.3162	34.6	70.9	1.3565	37.8	77.4	1.3986	41.0
58.0	1.2781	31.3	64.5	1.3168	34.6	71.0	1.3572	37.9	77.5	1.3992	41.0
58.1	1.2787	31.4	64.6	1.3174	34.7	71.1	1.3578	37.9	77.6	1.3999	41.1
58.2	1.2793	31.4	64.7	1.3180	34.7	71.2	1.3585	38.0	77.7	1.4005	41.1
58.3	1.2799	31.5	64.8	1.3186	34.8	71.3	1.3591	38.0	77.8	1.4012	41.2
58.4	1.2804	31.5	64.9	1.3192	34.8	71.4	1.3597	38.1	77.9	1.4019	41.2
58.5	1.2810	31.6	65.0	1.3198	34.9	71.5	1.3604	38.1	78.0	1.4025	41.3

RELATION OF BRIX, SPECIFIC GRAVITY, AND BAUMÉ—(*Concluded*).

Per Cent of Sugar.	Specific Gravity.	Degree Baumé.	Per Cent of Sugar.	Specific Gravity.	Degree Baumé.	Per Cent of Sugar.	Specific Gravity.	Degree Baumé.	Per Cent of Sugar.	Specific Gravity.	Degree Baumé.
78.1	1.4032	41.3	80.1	1.4165	42.3	82.1	1.4300	43.3	84.1	1.4437	44.2
78.2	1.4039	41.4	80.2	1.4172	42.3	82.2	1.4307	43.3	84.2	1.4443	44.3
78.3	1.4045	41.4	80.3	1.4179	42.4	82.3	1.4314	43.4	84.3	1.4450	44.3
78.4	1.4052	41.5	80.4	1.4185	42.4	82.4	1.4320	43.4	84.4	1.4457	44.3
78.5	1.4058	41.5	80.5	1.4192	42.5	82.5	1.4327	43.5	84.5	1.4464	44.4
78.6	1.4065	41.6	80.6	1.4199	42.5	82.6	1.4334	43.5	84.6	1.4471	44.4
78.7	1.4072	41.6	80.7	1.4205	42.6	82.7	1.4341	43.5	84.7	1.4478	44.5
78.8	1.4078	41.7	80.8	1.4212	42.6	82.8	1.4348	43.6	84.8	1.4485	44.5
78.9	1.4085	41.7	80.9	1.4219	42.7	82.9	1.4354	43.6	84.9	1.4492	44.6
79.0	1.4092	41.8	81.0	1.4226	42.7	83.0	1.4361	43.7	85.0	1.4498	44.6
79.1	1.4098	41.8	81.1	1.4232	42.8	83.1	1.4368	43.7	85.1	1.4505	44.7
79.2	1.4105	41.9	81.2	1.4239	42.8	83.2	1.4375	43.8	85.2	1.4512	44.7
79.3	1.4112	41.9	81.3	1.4246	42.9	83.3	1.4382	43.8	85.3	1.4519	44.8
79.4	1.4119	42.0	81.4	1.4253	42.9	83.4	1.4388	43.9	85.4	1.4526	44.8
79.5	1.4125	42.0	81.5	1.4259	43.0	83.5	1.4395	43.9	85.5	1.4533	44.9
79.6	1.4132	42.1	81.6	1.4266	43.0	83.6	1.4402	44.0	85.6	1.4540	44.9
79.7	1.4138	42.1	81.7	1.4273	43.1	83.7	1.4409	44.0	85.7	1.4547	45.0
79.8	1.4145	42.2	81.8	1.4280	43.1	83.8	1.4416	44.1	85.8	1.4554	45.0
79.9	1.4152	42.2	81.9	1.4287	43.2	83.9	1.4423	44.1	85.9	1.4561	45.1
80.0	1.4158	42.2	82.0	1.4293	43.2	84.0	1.4430	44.2	86.0	1.4568	45.1

polarizing in a 100-mm. tube (half the standard length) and multiplying the reading by 2. The clarifier best adapted as a rule for molasses and golden syrup is subacetate of lead.*

The Process.—The normal weight, 26.048 grams, of the molasses or syrup is dissolved in water in a 100-cc. flask, and in the case of molasses and "golden," or "drip" syrup, sufficient subacetate of lead solution is added to precipitate the coloring matter. From 5 to 10 cc. of the clarifier usually suffice. The flask is then filled to the mark with water and the contents shaken thoroughly and filtered. If on account of air bubbles it is difficult to make up to the mark, the bubbles may usually be dispelled by a drop of ether. With maple syrup no clarifier is, as a rule, necessary, though sometimes alumina cream is helpful. With a very



clear the solution, if the turbidity is due to carbonate. If cloudiness in the filtrate persists, weigh out a fresh portion of the sample, dilute, and add first the lead subacetate solution, and afterwards enough of a strong solution of sodium sulphate or common salt to precipitate the excess of lead; then fill to the mark and filter. Polarize, and conduct the inversion as directed on p. 483, using, however, a 100-mm. tube, and multiplying the reading by 2, both direct and invert.* Use Clerget's formula for calculation of the sucrose.

For medium- or light-colored grades of molasses, which yield but a small precipitate with lead subacetate, the above method of simple polarization, both direct and invert, gives results sufficiently accurate for ordinary work. For dark-colored, or "black-strap" molasses, or wherever extreme accuracy is required, the double dilution method of Wiley should be employed, which makes due allowance for the volume of the precipitate.

Double Dilution Method.†—Take half the normal weight of the sample and make up the solution to 100 cc., using the appropriate clarifier. Take the normal weight of the sample and make up a second solution with the clarifier to 100 cc. Filter and obtain direct polariscopic readings of both solutions. Invert each in the usual manner and obtain the invert reading of the two.

The true direct polarization of the sample is the product of the two direct readings divided by their difference. The true invert polarization is the product of the two invert readings divided by their difference.

Determination of Raffinose in Beet Sugar Molasses.—For the determination of sucrose and raffinose when present in the same solution, use the following formulas of Clerget:‡

$$\text{Sucrose} = \frac{C - 0.493a}{0.827} \quad \ldots\ldots\ldots \quad (1)$$

and

$$\text{Raffinose} = \frac{a - S}{1.85}, \quad \ldots\ldots\ldots \quad (2)$$

where a = direct reading, C = the algebraic sum of the direct and invert reading, and S = per cent of sucrose.

* The short tube (100 mm.) is preferred for polarizing molasses, not only on account of the more or less deep color of the clarified solution, but also because a molasses sample containing considerable commercial glucose would not read within the scale limits, if the 200-mm. tube were employed.

† Wiley and Elwell, Analyst, 1896, 21, p. 184.

‡ Spencer, Handbook for Chemists of Beet Sugar Houses and Seed-culture Farms.

Davoll * recommends for purposes of clarification of the molasses the use of powdered zinc after inversion of the molasses sample according to Clerget's method. He adds 1 gram of the zinc to the sample after inversion while at the temperature of 69° C., allowing it to act for three to four minutes at that temperature, after which he cools and filters, with the production of an almost colorless solution.

Determination of Reducing Sugar.—(*Estimated as Dextrose.*)—Dilute 5 grams of molasses or syrup with water in a 100-cc. graduated flask, using 2 to 5 cc. of subacetate of lead solution. Make up to 100 cc., filter, take an aliquot part of the filtrate (25 to 50 cc.) and make this up to 100 cc., the amount taken being such that, when diluted, the solution will contain not more than ½% of dextrose. If lead subacetate has been used to clarify, add to the aliquot part taken and before dilution, enough sodium sulphate to precipitate the excess of lead, then filter and make up to the 100 cc. mark.

Determine the reducing sugar in this solution by either volumetric or gravimetric Fehling processes.

U. S. Standard Molasses is molasses containing not more than 25% of water, nor more than 5% of ash.

Adulteration of Molasses and Syrups.—A common adulterant of all these products is commercial glucose. From its water-white color and inert sweetness, no less than from its cheapness, it forms an admirable adulterant for dark-colored or low-grade molasses and syrups, counteracting to a great extent by its smoothness the strong and often disagreeable taste of the inferior products with which it is mixed. Thus a grade of molasses too cheap to be ordinarily used for food purposes can be made to assume the appearance, and to some extent the taste, of the higher-priced and light-colored grades by admixture with commercial



or absence of commercial glucose can usually be established. The direct polarization of a normal solution of pure molasses should not be much in excess of 50° on the Soleil-Ventzke scale, while a pure, dark-colored molasses should polarize well under 40°. Golden syrup and maple syrup read higher than molasses, and a normal solution of pure maple syrup may have a direct polarization as high as 65°, being more often than not above 60°.

An excessively high direct polarization is at once an indication of the presence of commercial glucose, while an invert reading at ordinary room temperature to the right of the zero-point is an almost positive proof of its presence in either of the above products.

The optically active constituents of commercial glucose, viz., dextrin, maltose, and dextrose, are present in such varying amounts, that it is impossible to determine accurately the exact amount of this adulterant in complex saccharine products which themselves contain components common to glucose. Its approximate amount can, however, be very satisfactorily estimated in molasses and syrups by the use of the following formula:

$$G=\frac{(a-S)100}{175},*$$

where G=per cent of commercial glucose, a=direct polarization, and S=per cent of cane sugar previously obtained from the Clerget formula (p. 483). A large amount of invert sugar present affects the accuracy of this formula. It is especially applicable to maple syrup, wherein the per cent of invert sugar is small, but may be applied also to molasses and golden syrup, wherein the amount of invert sugar is not so large but that results may be obtained generally within 2 or 3% of the truth.†

In saccharine products containing considerable invert sugar the method described on page 514 should be used.

* Leach, U. S. Dept. of Agric., Bur. of Chem., Bul. 65, p. 48.

† The grade of glucose commonly employed by molasses and syrup manufacturers for admixture with their products, is the 42°-Bé. variety. A grade of greater density could evidently not be used directly, since it would not readily flow. Indeed this grade (42° Bé.) is sometimes thinned by water before mixing with the thinner syrups. The factor 175 is the one selected, because this is about the maximum polarization of the 42°-Bé. grade, and it is right to give the benefit of any reasonable doubt to the manufacturer.

For the sake of greater precision the result might well be expressed "in terms of commercial glucose polarizing at 175°."

TYPICAL ANALYSES OF MOLASSES AND SYRUPS ADULTERATED WITH COMMERCIAL GLUCOSE.

	Polarization.			Per Cent Sucrose (Clerget's Formula).	Reducing Sugar (Dextrose).	Commercial Glucose (Leach's Formula).	Moisture.	Ash.
	Direct.	Invert.	Temperature.					
(*a*) Molasses.....	62	+36.3	18°	19	30.03	24.6	29.36	3.83
(*b*) "	98.7	+71.9	18°	19.9	27.62	45.0	27.98	3.53
(*c*) "	109.7	+90	17°	14.5	33.11	54.4	22.02	2.67
(*a*) Golden drip syrup..	73.5	+39.8	18°	25	31.61	27.7	23.67	3.94
(*b*) " " " ..	109.4	+87.6	17°	16.9	33.44	52.8	24.48	2.51
(*c*) " " " ..	143.6	+136.0	18.4°	5.6	38.17	78.5	21.52	1.00
(*a*) Maple syrup.....	76.3	+7.6	18.6°	51	10.55	14.4	31.91	0.65
(*b*) " "	77.9	+24	19°	40.1	.....	21.6	23.44	
(*c*) " "	87.0	+30.6	22.4°	42.5	16.90	25.4	28.80	1.08

Determination of Dextrin. — According to Beckman's method a weighed amount of the honey or molasses is diluted with an equal volume of water and from ten to twelve times its volume of methyl alcohol is added. The precipitated dextrin is collected in a tared filter and thoroughly washed with methyl alcohol, after which it is dried and weighed.

Reduction of Saccharine Products to an Ash for Mineral Analysis. —If a considerable quantity of molasses, syrup, or other saccharine substance is to be burnt to an ash, it is both tedious and annoying to ignite directly, by reason of the excessive swelling and frothing of such substances during ignition. Small quantities of molasses, syrup, or honey may with care be reduced to an ash by the method described on page 481.

If a readily controlled electric current is available, it may be utilized as follows:* Mix 100 grams of molasses, syrup, or other saccharine solution, which should be evaporated to syrupy consistency if not already

the mass is reduced to a fine, dry char, which may then be readily burnt to a white ash in the original dish over a free flame or in a muffle.

Or, 100 grams of the molasses or syrupy solution to be ashed may be first evaporated to dryness and afterward mixed with from 10 to 20 cc. of concentrated sulphuric acid in a porcelain evaporating-dish, or if the substance to be ashed be a dry sugar or confectionery, 20 grams are mixed with the above amount of acid. Heat is gently applied by means of the gas flame till the swelling and frothing have ceased, which usually requires only a few minutes. The final ignition is then accomplished in the usual manner, nitric acid being added if necessary to completely destroy the organic matter.

Determination of Tin in Molasses.—Fuse the ash from a weighed portion of the sample with sodium hydroxide in a silver crucible, dissolve in water, and acidulate with hydrochloric acid; filter and precipitate the tin from this solution with hydrogen sulphide; wash the precipitate on a filter and dissolve it in an excess of ammonium sulphide. Filter this solution into a tared platinum dish, and deposit the tin directly in the dish by electrolysis, using a current of 0.05 ampere and the apparatus described on page 493.

Distinction between Invert Sugar, Maltose, and Lactose.*—All these sugars reduce Fehling's solution. Dextrose and levulose (invert sugar) when boiled with Barfoed's copper acetate solution (14 grams crystallized copper acetate and 5 cc. acetic acid in 200 cc. water) will form a precipitate of cuprous oxide, while neither maltose nor lactose will do this. The solution, which has thus been tested for invert sugar and found to be free, or the filtrate from the cuprous oxide precipitate, is treated with an excess of basic lead acetate, filtered, and to the filtrate is added an excess of sodium sulphate solution to precipitate the lead. The solution is again filtered and treated with copper sulphate solution, if not already blue. It is then made alkaline with sodium hydroxide and heated to boiling. A red precipitate of cuprous oxide at this stage indicates either lactose or maltose or both.

A solution of the sugar, made strongly ammoniacal, is then mixed with alkaline bismuth solution † and the container is set in a water-bath at 60° C. Maltose soon reduces the bismuth, but lactose does not.

To test for lactose, add strong nitric acid to the solid sugar residue

* Bartley and Mayer, Merck's Report, 12 (1903), p. 100.

† This reagent is prepared as follows: Bismuth subnitrate, 2 grams; Rochelle salt, 4 grams; sodium hydroxide, 8 grams; dissolved in 100 cc. of water by the aid of heat.

and warm gently till red fumes come off. Then set the container in hot water and cool gradually. Crystals of mucic acid appear after a time if any appreciable amount of lactose be present.

Determination of Lactose or Maltose.—Either sugar, if in solution free from other reducing sugars, may be determined by the volumetric Fehling method (p. 486) or by the Defren method, using the table on page 490.

For the determination of maltose in commercial glucose, see page 509.

Estimation of Cane Sugar and Dextrose in Mixtures.—Obtain true direct and invert readings of a normal solution of the mixture. Determine the per cent of sucrose by Clerget's formula (p. 483). This figure represents the right-handed rotation due to sucrose. Subtracting this from the direct polarization, the difference represents the right-handed rotation due to dextrose. The specific rotary power of sucrose is 66.5 and that of dextrose 52.3.

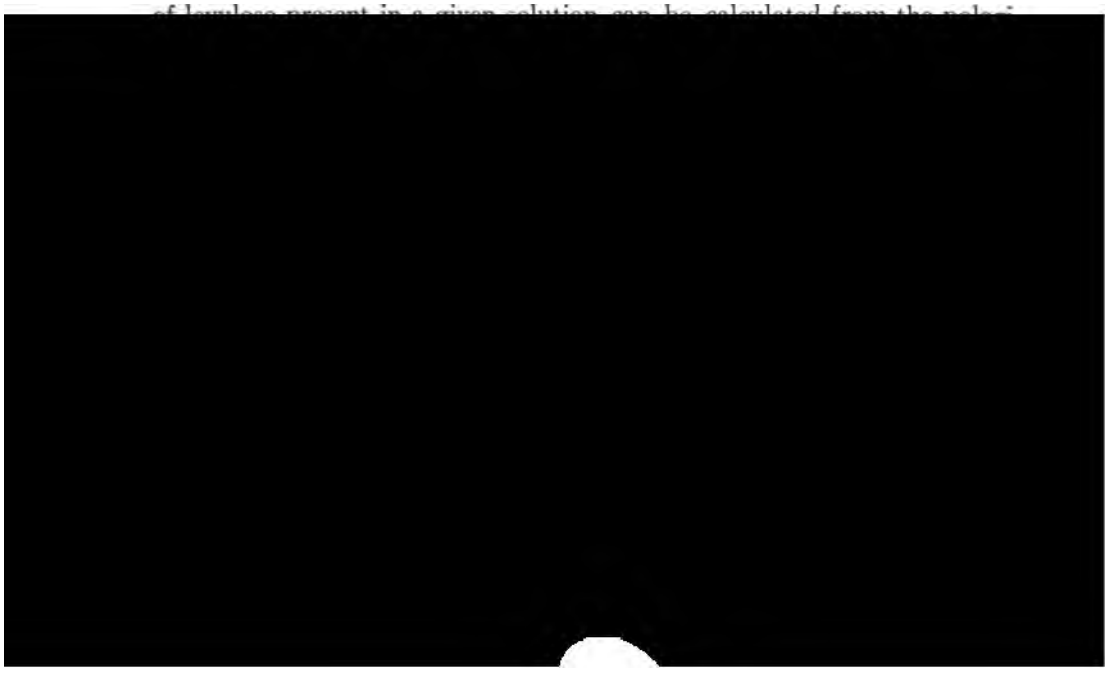
Calling d the percentage of dextrose and R' the right-handed rotation due to dextrose as above obtained, if the Soleil-Ventzke scale is used,

$$66.5 : 52.3 = d : R',$$

whence

$$d = \frac{66.5R'}{52.3}.$$

Determination of Levulose.*—On page 484 attention was called to the variation in the rotary power of levulose with the temperature. This variation is constant, and 1 gram of levulose in 100 cc. of water produces a decrease in left-handed reading of 0.0357° on the cane sugar (Ventzke) scale for each 1° C. increase in temperature. Therefore, the weight



If L' = percentage of levulose,
L = weight of levulose in solution,
W = weight of sugar sample made up to 100 cc.,

$$L' = \frac{L \times 100}{W}.$$

In a normal solution $W = 26.048$.

Analysis of Commercial Glucose.—Wiley * has worked out a method for calculating the percentage of dextrin, maltose, and dextrose present in commercial glucose, based on the specific rotary power of these substances and on the relative reducing power of maltose and dextrose. To apply this method, the operator, if he has a polariscope reading in sugar scale degrees, must ascertain the equivalent readings in angular degrees from the table on page 478, and calculate the specific rotary power in each case from the formula $(\alpha)_D = \frac{100a}{cl}$, page 479.

Thus, if he possesses a Schmidt and Haensch instrument, he should multiply the true reading, as obtained on that instrument, with a normal solution of the given sugar or mixture, by the factor 0.3468, to convert the reading into circular degrees from which to figure the specific rotary power as above.

The specific rotary power of dextrin is fixed at 193, that of maltose 138, and that of dextrose at 53.

Then if P = total polarization of the mixture in terms of specific rotary power, d = per cent dextrose, m = per cent maltose, and d' = per cent dextrin,

$$P = 53d + 138m + 193d'. \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad (1)$$

The value of P is obtained from observation and calculation as above described on a known solution of the sample, say 10 grams in 100 cc. The reducing sugars, maltose and dextrose, are then removed, preferably by oxidation with cyanide of mercury, as follows: †

Prepare the reagent by dissolving 120 grams mercuric cyanide and 120 grams sodium hydroxide in water, mixing the two solutions, and making up to 1,000 cc. Remove any precipitate that may gather by filtration.

Make a solution of 10 grams of the glucose sample in 100 cc. and take 10 cc. of this solution in a 50-cc. graduated flask. Add sufficient

* Chem. News, 46, p. 175; Agric. Anal., 3, pp. 288–290.
† Wiley, Agric. Analysis, p. 290.

mercuric cyanide solution to have an excess of reagent after the oxidation (from 20 to 25 cc.), and boil for three minutes under a hood with a good draft. Cool and neutralize the alkali with concentrated hydrochloric acid, adding the latter till the brown color is discharged. By this method the optical activity of the maltose and dextrose is discharged, while that of the dextrin remains unaffected. From the polariscope reading calculate as above the specific rotary power of the dextrin (P'). Then

$$P' = 193d'. \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad (2)$$

The reducing power on Fehling's solution of dextrose is to that of maltose as 100 is to 62. Whence, if R = reducing sugar (reckoned as dextrose) we have

$$R = d + 0.62m. \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad (3)$$

Subtracting equation (2) from equation (1) we have

$$P - P' = 53d + 138m. \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad (4)$$

Multiplying equation (3) by 53 and subtracting from equation (4),

$$\begin{aligned} P - P' &= 53d + 138m, \\ 53\ R &= 53d + 32.86m, \\ P - P' - 53R &= 105.14m. \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad (5) \end{aligned}$$

Therefore

$$m = \frac{P - P' - 53R}{105.14}, \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad (6)$$

$$d = R - 0.62m, \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad (7)$$

$$d' = \frac{P'}{193}. \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad (8)$$



Arsenic in Commercial Glucose.—Like all products wherein commercial sulphuric acid is employed in its manufacture, glucose sometimes contains arsenic, though usually in minute traces. Arsenic is readily indicated, when present, by the Gutzeit test, conducted as follows: 2 grams of the sample are introduced into a small Erlenmeyer flask of about 100 cc. capacity, and diluted with 5 to 10 cc. of water. Scraps of arsenic-free, granulated zinc are then added. A small filter-paper is carefully folded smoothly around the bottom of a cork that loosely fits the mouth of the flask, and is moistened with a concentrated solution of mercuric chloride. From 6 to 8 cc. of arsenic-free concentrated hydrochloric * or sulphuric acid are then added to the flask, so as to produce rapid, but not too violent evolution of gas, and the cork is loosely inserted.

After ten minutes the cork is removed, and, if a yellow stain is present on the filter, arsenic is indicated. The amount of arsenic present varies with the depth of color, and if a large amount is present the stain may be dark brown or even black.

Sulphides interfere with the Gutzeit test, but are rarely present in commercial glucose. Unless sure of the purity of the reagents it is well to make a blank test thereon. In such a blank, the filter should be perfectly white after ten minutes.

The amount of arsenic may be roughly determined colorimetrically by the Gutzeit method.

For more careful determination, employ the Marsh apparatus, into which the diluted glucose may be directly introduced without previous treatment.

HONEY.

Composition and Occurrence.—Honey is the saccharine product deposited by the bee (*Apis mellifica*) in the cells of honeycomb, which the insect forms out of wax secreted by its body. Honey has its source chiefly in the nectaries of flowers, from which the bees abstract it. The juices of ripe fruits and the sap of trees also furnish honey. During the secretion of the honey in the body of the bee, the sucrose, which forms the chief constituent of the fruit juice or nectar, becomes for the most part inverted, forming, in the honey, dextrose and levulose.

The flavor of honey varies considerably according to its source. Besides water, the sugars, and mineral matters, pollen is usually present,

* Hydrochloric acid is better than sulphuric acid, as the action is much more brisk with pure zinc.

derived from the flowers, also as a rule a small quantity of wax, and nearly always appreciable amounts of various organic acids, such as formic.

A large number of samples of genuine honey analyzed in 1897 for the Department of Inland Revenue, Canada (Bul. 47), showed the following variations:

Direct polarization	− 2.4	to	−19
Invert "	−10.2	"	−28
Sucrose (by Clerget)	0.5%	"	7.64%
Invert sugar	60.37%	"	78.8%
Water	12%	"	33%
Ash	0.03%	"	0.50%

The following are typical analyses of honey adulterated with cane sugar:

	A.	B.	C.
Direct polarization	+34.7	+12	+ 1.2
Invert "	−24	−17.6	−21.5
Temperature	14°	15°	19.5°
Sucrose (Clerget)	43.16%	21.8%	17.07%
Invert sugar	42.48%	60.03%	67.2%
Water	42.42%	21.15%	15.56%
Ash	.....	.11%	0.06%

The following are typical analyses of honey adulterated with commercial glucose:

	A.*	B.	C.
Direct polarization	+147	+66.9	+101.5



In the case of comb honey, cut with a knife across the top of the comb, if sealed, and separate completely from the comb by straining through a 40-mesh sieve.

Water and Ash are determined as directed on page 498 in the case of molasses and syrup.

For polarization of honey both direct and invert, proceed as directed on page 502 except that instead of subacetate of lead, alumina cream is employed for a clarifier, the reagent being added in excess before making up to the mark in the 100-cc. flask. On account of birotation, a drop or two of ammonia is added.* Aside from these precautions exactly the same method of procedure is adopted as in the case of molasses and syrup.

Determination of Invert Sugar.—Weigh 5 grams of the sample into a 100-cc. graduated flask, dissolve in water, clarify with about 5 cc. of alumina cream, and make up to the 100-cc. mark. Filter and determine invert sugar in an aliquot part of the filtrate by either volumetric or gravimetric Fehling processes.

Adulteration of Honey.—The most common adulterants of honey are cane sugar and commercial glucose. Sometimes both are employed in the same sample. Gelatin is also said to be used. It appears to be a fact that bees may be made to feed upon cane syrup or commercial glucose, if these materials are placed in proximity to their hives, so that in some instances the adulterant may be supplied through the medium of the bee. Sophisticated honey is often put up in tumblers or jars containing pieces of honeycomb, so that presence of the comb is by no means proof of its purity. Sometimes the comb itself is artificial.† Comb-honey, sold in the frame as sealed by the bees, is never adulterated, except when the bees are fed upon glucose or cane sugar.

Gelatin is indicated if a precipitate occurs in the diluted sample with a solution of tannic acid.

A right-handed polarization of honey at once makes it suspicious. The author has never found a sample of genuine honey that polarized to the right of the zero-point, though cases are on record of pure right handed honeys. Such are said to have been yielded from the exudation of certain pine trees, the honey from this source possessing notable quan-

* Frühling, Zeits. öffentl. Chemie, 4 (1898), p. 410.

† A sample of alleged honey was purchased by one of the inspectors of the Food and Drug Department of the Mass. State Board of Health, put up in a glass jar with a large mass of honeycomb and a dead bee. No genuine honey was found, the mixture consisting of commercial glucose and cane sugar. Even the comb was artificial.

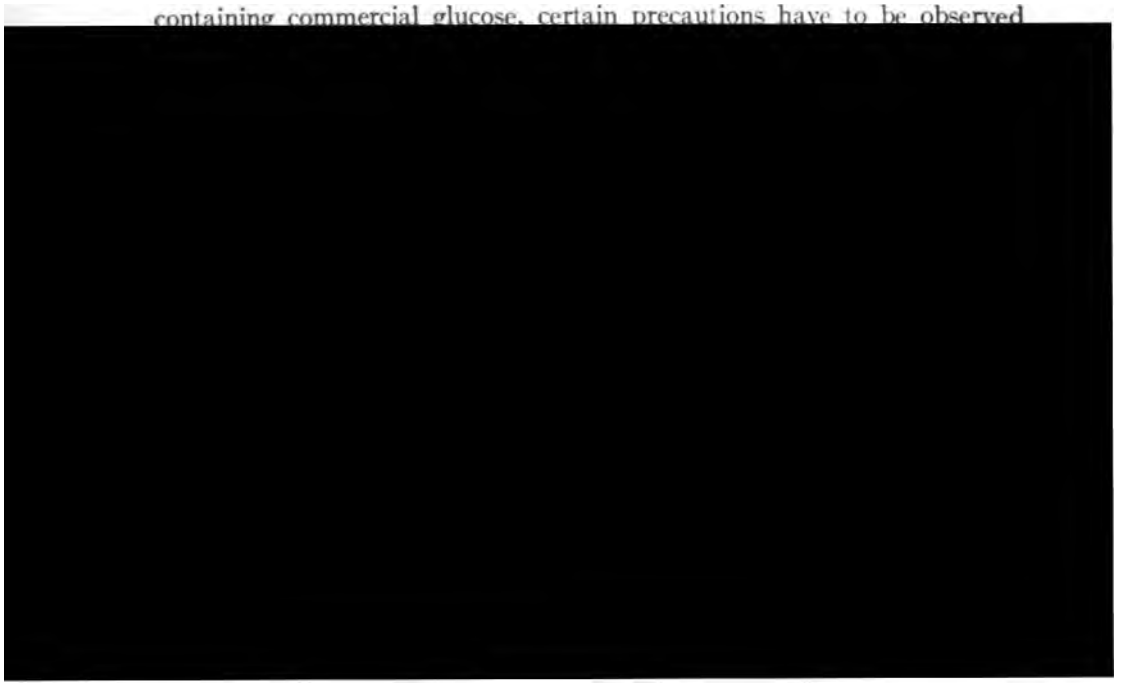
tities of dextrin, which in normal honey is absent. A high right-handed rotation points either to cane sugar or to commercial glucose as adulterants. If, after inversion of the honey sample, the polarization is left-handed, the adulterant is undoubtedly cane sugar. If the polarization after inversion is still right-handed, commercial glucose is no doubt present.

Notable amounts of calcium sulphate, when tested in the diluted honey, indicate commercial glucose. The test for calcium is made with ammonia and ammonium oxalate; that for sulphuric acid with hydrochloric acid and barium chloride in the usual manner.

The presence of commercial glucose is strongly indicated if, on the addition of 3 or 4 volumes of strong alcohol to the honey, a precipitate of dextrin is found. Pure honey should show only a slight milkiness and no actual precipitate when thus treated.

Determination of Commercial Glucose in Honey.—Except for rough work, the method described on page 505 for calculating the per cent of commercial glucose from the sucrose and from the direct polarization is not recommended for use with honey and other products wherein the invert sugar is so large as to considerably affect its accuracy. In this case, it is best after inversion to polarize the sample at 87° C., the temperature at which the reading due to invert sugar would be 0. At this temperature, any right-handed polarization can be accounted as due to commercial glucose.

As in the case of molasses, the writer advocates assuming 175° as the direct polarization of the glucose used, this being about the maximum reading for a normal solution of 42°-Bé. glucose. Lythgoe has shown that in polarizing at high temperatures samples of saccharine products containing commercial glucose, certain precautions have to be observed



the invert reading, weigh out a separate half-normal portion in a 100-cc. sugar-flask, dilute to about 70 cc. with water, add 7 cc. of hydrochloric acid (specific gravity 1.20), and heat in the regular manner to 68° C. Cool, add a few drops of phenolphthalein, and neutralize with sodium hydroxide. Add a few drops of dilute hydrochloric acid to discharge the pink color, then from 5 to 10 cc. of the appropriate clarifier (in the case of honey, alumina cream), cool again, and make up to the 100-cc. mark. Polarize in a 200-mm. jacketed tube at 87°, and multiply the reading by 2. The invert reading may be obtained on the same solution at 20°, if desired. Divide the true reading at 87° by 163 * and multiply the result by 100 for the percentage of commercial glucose in terms of glucose polarizing at 175°. See also Appendix, page 769.

Polarization at the temperature of 87° can most readily be effected by the use of a water-jacketed tube, as shown in Fig. 110. An all-metal tube, the interior of which is heavily gold-plated to avoid corrosion by acid, is preferable to one in which the inner tube is glass with a metal jacket, as in the latter leaky joints are liable to occur, due to uneven expansion. A tubulure is provided in the outer tube for a thermometer, so that the exact temperature may be noted. A tank of boiling water placed on a shelf above the polariscope is connected by rubber tubing with the jacketed tube as it rests in the polariscope, as shown in Fig. 110.

BEESWAX.—The purity of beeswax is best established by determining its melting-point, its specific gravity, its saponification equivalent, and its refractometric reading. The melting-point of pure wax is about 64° C., while that of paraffin, its chief adulterant, is from 52° to 55° C. Its saponification equivalent should be from 87.8 to 107, while that of paraffin is 0.

Method of Determining Specific Gravity of Beeswax.†—Place a weighed rod of the wax, about 1 to 1.5 cm. long by 0.5 cm. diameter, in an accurately marked 50-cc. flask, and run in water from a burette till the water level reaches the mark. 50 cc. minus the burette reading represent the volume occupied by the wax. The rod should be made to lie flat on the bottom of the flask, so that the incoming water will force its ends against the sides and prevent the end from rising above the mark. The volume

* The true polarization at 87° C. of a normal solution of glucose subjected to inversion and neutralization as above (but without the use of the clarifier), will be about 93% that of the direct polarization of the sample in the cold. Hence $175 \times 0.93 = 162.7$.

† Bulletin 13, U. S. Department of Agriculture, Division of Chemistry, p. 842.

of the rod, divided by its weight, gives its specific gravity. The specific gravity of various mixtures of wax of 0.969 specific gravity and paraffin of 0.871 are given in the following table, prepared by Wagner, so that

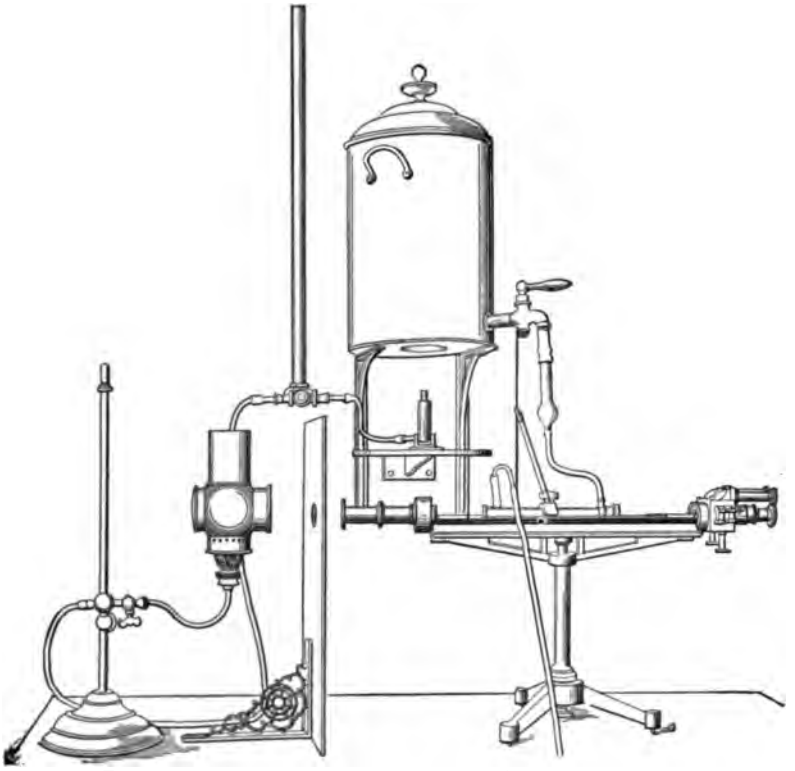


FIG. 110.—Apparatus for Polarizing at High Temperatures.

The Abbé refractometer may be used with perfect safety and, when available, is to be preferred for the examination of beeswax. Many food laboratories are, however, not equipped with the Abbé, but nearly all find the butyro-refractometer indispensable. The latter instrument was primarily designed for such substances as butter and lard, so that the manufacturers did not intend it to be subjected to as high a temperature as 65°. They have, however, assured the author that if care be taken to bring the temperature very slowly and gradually to the required degree, 65°, and to avoid also sudden cooling, the cement that secures the prisms in place will not be appreciably affected; otherwise cracking or loosening of the cement would be liable to occur after a time.

At 65° C. pure beeswax should have a reading on the butyro-refractometer of 30 to 31.5,* while that of paraffin is from 11 to 14.5.†

CONFECTIONERY.

The composition of confectionery is more complex than that of the saccharine products hitherto considered. As a rule, cane sugar, or one of its products, as molasses, forms the basis of most of the confections. Commercial glucose is also a common ingredient, while a large variety of such materials as eggs, butter, chocolate, various flavoring extracts, spices, nuts, and fruits, enter into the composition of confectionery.

U. S. Standard Candy is candy containing no terra alba, barytes, talc, chrome yellow, or other mineral substances or poisonous colors or flavors, or other ingredients injurious to health.

Adulteration.—Of late the adulteration of confectionery has been held largely in check by the National Confectioners' Association of the United States, which has fixed high standards of purity, and has been very zealous in restricting the use of harmful adulterants.

Commercial glucose is not regarded as an adulterant of confectionery by the above-named association and by but few of the state laws. On the contrary, any ingredient, other than color, that has no food value, may logically be considered as an adulterant. Under this head are included such substances as paraffin, as well as make-weight mineral matters, such as terra alba, talc, or calcium sulphate.

Most of the actually harmful ingredients employed in confectionery have been inherent in the coloring matters, or in the alcohol or fusel oil used in the manufacture of brandy drops and allied confections.

* n_D, 1.4452 to 1.4463. † n_D, 1.4310 to 1.4335.

Colors in Confectionery.—A very wide range of colors is necessarily employed in the manufacture of confectionery, and the almost endless variety of coal-tar dyes now available lend themselves most readily to the confectioner's needs. Elsewhere, under "colors," lists of injurious and non-injurious dyes are given as compiled by the National Confectioners' Association, though it is not always readily apparent how the lines are drawn.

The tinctorial power of these dyes is so high that the actual amount of substance contained in a thin coating of the color on the outside of the candy is exceedingly small, so that it is doubtful whether serious cases of injury have ever arisen from their use.

Such was not the case formerly, before the prevalence of the coal-tar dyes, when such poisonous mineral pigments as chromate of lead were frequently used. Only one or two instances of the use of lead chromate in candy have come to the author's attention within ten years, since more satisfactory and harmless yellow colors among the azo-dyes are now obtainable.

ANALYSIS OF CONFECTIONERY.—The following have been submitted by the author as provisional methods of procedure for the A. O. A. C.:*

(1) **Products of Practically Uniform Composition Throughout.**—(*a*) *Lozenges and Other Pulverizable Products.*—Grind in a mortar or mill to a fine powder. For *total solids*, weigh from 2 to 5 grams of the powdered sample in a tared platinum dish, and dry in a McGill oven to constant weight.

For *Ash*, ignite the residue from total solids in the original dish, observing the precautions given under sugar (p. 481), and molasses (p. 506).

(*b*) *Semi-plastic, Syrupy, or Pasty Products.*—Weigh 50 grams of the sample into a 250-cc. graduated flask, mix thoroughly or dissolve,



(3) **Sugar-coated Fruit, Nuts, etc.**—In case of a saccharine coating inclosing fruit, nuts, or any less readily soluble material, dissolve or wash off the exterior coating in water, which may, if desired, be evaporated to dryness for weighing, and proceed as in (1).

(4) **Candied or Sugared Fruits.**—Proceed as in the examination of fruits (Chapter XIX).

Detection of Mineral Adulterants.—As in the case of molasses, a considerable quantity, say 100 grams, should be reduced to an ash for examination for mineral adulterants, such as talc, calcium sulphate, and iron oxide, which are detected by regular qualitative tests.

Detection of Lead Chromate.—Fuse the ash in a porcelain crucible with a mixture of sodium carbonate and potassium chlorate, boil the fused residue with water, neutralize with acetic acid, filter, and treat the filtrate with barium chloride or lead acetate solution. A yellow precipitate indicates a chromate. Treat the insoluble part of the fusion with nitric acid, and test for lead in the usual manner.

If a drop of ammonium sulphide be applied to a piece of confectionery colored with lead chromate, it will produce a black coloration.

Determination of Ether Extract.—The ether extract includes the fat derived from chocolate, eggs, or butter, as well as any paraffin present. Measure 25 cc. of the 20% solution (1) (*b*) (p. 518) into a very thin, readily frangible glass evaporating-shell (*Hoffmeister's Schälchen*), containing 5 to 7 grams of freshly ignited asbestos fiber; or, if impossible to thus obtain a uniform sample, weigh out 5 grams of the mixed, finely divided sample into a dish, and wash with water into the asbestos in the evaporating-shell, using, if necessary, a small portion of the asbestos fiber on a stirring-rod to transfer the last traces of the sample from dish to shell. Dry to constant weight at 100°, after which cool, wrap loosely in smooth paper, and crush into rather small fragments between the fingers, carefully transferring the pieces with the aid of a camel's-hair brush to an extraction-tube, or to a Schleicher and Schull cartridge for fat extraction. Extract with anhydrous ether or with petroleum ether in a continuous extraction apparatus for at least twenty-five hours. Transfer the solution to a tared flask, evaporate the ether, dry in an oven at 100° C. to constant weight, and weigh.

Determination of Paraffin.—Add to the ether extract in the flask, as above obtained, 10 cc. of 95% alcohol, and 2 cc. of 1:1 sodium hydroxide solution, connect the flask with a reflux condenser, and heat for an hour on the water-bath or until saponification is complete. Remove the con-

denser, and allow the flask to remain on the bath till the alcohol is evaporated off, and a dry residue is left. Treat the residue with about 40 cc. of water, and heat on the bath, with frequent shaking, till everything soluble is in solution. Wash into a separatory funnel, cool, and extract with four successive portions of petroleum ether, which are collected in a tared flask or capsule. Remove the petroleum ether by evaporation, and dry in the oven to constant weight.

It should be noted that any phytosterol or cholesterol present in the fat would come down with the paraffin, but the amount would be so insignificant that, except in the most exacting work, it may be disregarded. The character of the final residue should, however, be confirmed by determining its melting-point and specific gravity, and by subjecting it to examination in the butyro-refractometer. The melting-point of paraffin is about 54.5° C.; its specific gravity at 15.5° C. is from 0.868 to 0.915, and on the butyro-refractometer the reading at 65° C. is from 11 to 14.5.

Determination of Starch.—Measure gradually 25 cc. of a 20% aqueous solution or uniform mixture of the sample into a hardened filter or Gooch crucible, or transfer by washing 5 grams of the finely powdered substance to the filter or Gooch, and allow the residue on the filter to become air-dried. Extract with five successive portions of 10 cc. of ether, then wash with 150 cc. of 10% alcohol, and finally with 20 cc. of strong alcohol. Transfer the residue to a large flask and boil gently for four hours with 200 cc. of water and 20 cc. of hydrochloric acid (specific gravity 1.125), the flask being provided with a reflux condenser. Cool, neutralize with sodium hydroxide, add 5 cc. of alumina cream, and make up the volume to 250 cc. with water. Filter and determine the dextrose in an aliquot part of the filtrate by any of the various Fehling methods. The weight of the dextrose multiplied by 0.9 gives the weight of the starch.

Commercial glucose is roughly determined by polarizing the sample at 87° C., as in the case of honey (p. 514).

Confectionery is made in such a wide variety of forms, and these differ in consistency to such an extent that commercial glucose of all available degrees of density can be utilized to advantage in one product or another. In this respect confectionery is unlike honey and molasses, wherein a fairly uniform grade of commercial glucose is necessarily used for mixing, this grade being naturally selected with reference to its similarity in density to the molasses. On this account the glucose factor used for honey and molasses (175) may in some varieties of confectionery be too high.

Determination of Alcohol in Syrups Used in Confectionery.—(*Brandy-drops.*)—Open each drop by cutting off a section with a sharp knife, and collect in a beaker the syrup of from 15 to 25 of the drops, which will usually yield from 30 to 50 grams of syrup. Strain the syrup into a tared beaker through a perforated porcelain filter-plate in a funnel to separate from particles of the inclosing shell, and ascertain the weight of the syrup. Wash into a distilling-flask, dilute with half its volume of water, and distil off into a tared receiving-flask a volume equal to the original volume of syrup taken. Ascertain the weight of the distillate and its specific gravity by means of a pycnometer. Multiply the per-centage by weight of alcohol corresponding to the specific gravity, as found in the tables on page 531 *et seq.*, by the weight of the distillate, and divide this by the weight of syrup taken. The result is the per cent by weight of alcohol in the syrup.

Detection of Colors.—It is sometimes necessary to macerate a considerable mass of the material to remove the color, which is, however, in the majority of cases readily soluble. The insoluble colors are nearly all mineral pigments to be looked for in the ash, as in the case of chromate of lead (p. 519). Frequently the coloring matter is confined to a thin outer layer, which is readily washed off.

The solution of the dyestuff is examined as directed under colors.

Detection of Arsenic.—Arsenic may be present through impure coloring-matter. If the color is confined to an exterior coating, this should be washed off and examined. If distributed through the mass, a solution of the whole should be taken. Examine for arsenic by the Gutzeit or Marsh method, as directed under glucose (p. 511).

REFERENCES ON SUGARS.

BABINGTON, F. W. Sugars, Syrups, and Molasses. Can. Inl. Rev. Dept., Bul. 25.
—— Maple Syrup. Can. Inl. Rev. Dept., Bul. 45.
BARTLEY, E. H., and MAYER, J. L. Identification of Carbohydrates. Merck's Report, 12, 1903, p. 100.
BROWN, H. T., MORRIS, G. H., and MILLAR, J. H. Experimental Methods employed in the Examination of the Products of Starch Hydrolysis by Diastase. Jour. Chem. Soc. Trans., 71 (1897), p. 72.
FRÄNKEL and HUTTER. Starch, Glucose and Dextrin. Phila., 1881.
FRESENIUS u. MAYRHOFER, J. Der Stärkesirup bei Zubereitung von Nahrungs- und Genussmitteln. Zeits. f. Unters. d. Nahr. u. Genussm., 1899, 21, 35 u. 279.
FRÜHLING, R. Anleitung zur Untersuchung der für die Zuckerindustrie. 6th ed. Braunsweig, 1903.
LANDOLT, H. Handbook of the Polariscope and its Practical Applications, 1882.
—— Trans. by Long, J. H. Optical Rotation of Organic Substances. Easton, 1902.
LEACH, A. E. The Determination of Commercial Glucose in Molasses, Syrups and Honey. Jour. Am. Chem. Soc., 25, 1903, p. 982.
—— Saccharine Products. U. S. Dept. of Agric., Bur. of Chem., Bul. 65, p. 43.
LOCK and NEWLANDS. A Handbook for Planters and Refiners. London, 1888.
MACFARLANE, T. Honey. Can. Inl. Rev. Dept., Bul. 45.
ROBIN, L. Sucrés. Analyse des Matières Alimentaires (Girard et Dupré), p. 525 Paris, 1894.
ROTH, H. L. A Guide to the Literature of Sugar. London, 1890.
SACHSSE, R. Die Chemie der Kohlenhydrate. Leipzig, 1877.
SHUTT, F. F., and CHARRON, A. T. Determination of Moisture in Honey. Trans. Royal Soc. Canada, 2d Series, 1902–3, 7, Section 3.
SIDERSKY, D. Traité d'Analyse des Matières Sucrées. Paris, 1890.
SPENCER, G. L. Handbook for Sugar Manufacturers and their Chemists. New York, 1897.
STEYDN, E. Die Untersuchung des Zuckers und Zuckerhaltiger Stoffe. Leipzig, 1893.
TOLLENS, B. Handbuch der Kohlenhydrate. Breslau, 1888.
TUCKER, J. H. Manual of Sugar Chemistry. New York, 1899.

CHAPTER XIV.

ALCOHOLIC BEVERAGES.

Alcoholic Fermentation.—In a broad sense all alcoholic liquors are saccharine products, in that they are essentially the result of the fermentation of sugar. In the case of fruits, the sugar already exists as such in their juices, which, when expressed, almost immediately on exposure to the air begin to undergo spontaneously the process of alcoholic fermentation, in accordance with the reaction:

$$(1)\quad \underset{\text{Dextrose or grape sugar}}{C_6H_{12}O_6} = 2\underset{\text{Alcohol}}{C_2H_6O} + 2\underset{\text{Carbon dioxide}}{CO_2}.$$

In the case of grains the process is more complex, involving a preliminary saccharous fermentation, whereby the starch is first transformed into sugar.

Thus

$$(2)\quad 2\underset{\text{Starch}}{C_6H_{10}O_5} + H_2O = \underset{\text{Dextrin}}{C_6H_{10}O_5} + \underset{\text{Dextrose}}{C_6H_{12}O_6}.$$

$$(3)\quad \underset{\text{Dextrin}}{C_6H_{10}O_5} + H_2O = \underset{\text{Dextrose}}{C_6H_{12}O_6}.$$

The process of alcoholic or vinous fermentation is largely dependent upon the presence of various species of yeasts, which either exist from the first in the expressed juices themselves, as in the case of wines, being derived from the skins of the grapes and from the air, or are introduced with some degree of selection, as in the case of beer.

In the juices of most fruits the sugar exists in the form of sucrose, mixed with variable amounts of invert sugar resulting from the inversion of the sucrose due to the action of ferments, such as invertase, a soluble ferment of yeast. The invert sugar nearly always predominates, and in some juices, as for instance the grape, nearly all the sugar has been inverted.

The above reaction, No. 1, illustrating the splitting up of grape sugar into alcohol and carbon dioxide, does not represent the practical yield of alcohol under ordinary conditions that occur in vinous fermentation, for, as a matter of fact, instead of 51.11 parts of alcohol and 48.89 parts carbon dioxide, which would theoretically result as above from the fermentation of 100 parts of dextrose, only about 95% of the theoretical yield can be obtained, so that in practice it is possible to form but about 48.5% alcohol and 46.5% carbon dioxide. The balance, amounting to some 5%, consists chiefly of glycerin, succinic acid, and traces of various compounds, including some of the higher-boiling alcohols (propyl, butyl, and amyl) and their ethers, which form the fusel oil of the distilled liquors.

Vinous fermentation takes place most readily in slightly acid liquids, at a temperature ranging from 25° to 30° C.

It is convenient to divide alcoholic beverages into two main groups, first the fermented and second the distilled liquors. The fermented liquors naturally subdivide themselves into (*a*) the products of the direct spontaneous fermentation of saccharine fruit juices, such, for example, as those of the apple and the grape, to form cider and wine respectively, and (*b*) the malted and brewed liquors, such as beer and ale, produced by the conversion of the starch of grain into sugar, and the final alcoholic fermentation of the latter.

The distilled liquors include such products as whiskey, brandy, rum, and gin, wherein alcoholic infusions prepared by previous fermentation in various ways are further subjected to distillation.

Alcoholic Liquors and State (or Municipal) Control.—The mere adulteration of liquors does not constitute the only feature which brings them within the scope of the public analyst's work and renders them



in the adulterated list as regards non-conformance to a standard of purity. While the raising of revenue is one purpose for the existence of these laws bearing on liquor license, an equally important object sought to be gained is doubtless the repression of intemperance.

Toxic Effects.—A popular impression seems to exist that the toxic effects of an adulterated liquor are far worse from a temperance standpoint than those of a sample of good standard quality, and it is a common experience of the public analyst to have submitted to him by well-meaning temperance advocates samples which are alleged to have caused the worst forms of intoxication, and are thus suspected of being impure. As a matter of fact the chief adulterants of liquors are water, sugar, and, in the case of beer, various bitter principles and vegetable extractives, none of which are on record as being in themselves actively toxic.*

Alcohol is the one ingredient of liquor which, more than any other, produces a marked physiological effect. Many liquors, especially those of the distilled variety classed as adulterated, are so considered by reason of their low alcoholic content through watering or otherwise, hence this commonest form of adulteration, far from being detrimental in itself, is actually helpful to the temperance cause.

Details of Liquor Inspection.—The same precautions should be carefully observed by officers making seizures of liquors for analysis, as by food inspectors, regarding safe delivery of the samples to the analyst. The following instructions are circulated by the State Board of Health of Massachusetts, which has in charge the inspection of liquors, concerning the taking of samples in that state and the transmission to the analyst:

DIRECTIONS FOR TAKING SAMPLES FOR ANALYSES.

The officer making a seizure, or taking samples of beer, should note at the time of such seizure the general appearance of the liquor,—as to whether it is clear or cloudy, whether it is still or has a strong head.

If the liquor is in bottles, take at least one pint bottle; if in barrels, draw a pint bottle from each. Request the owner to seal each sample taken. If the bottles have cork stoppers, cut the stoppers off level with the top of the bottle and cover with wax; if with patent stoppers, a little wax placed upon the wire at the point where it lays against the neck of the bottle is sufficient. If the owner refuses to seal it, then the officer

* The writer refers to substances intentionally added, and not to accidental impurities, such as arsenic, etc., that are occasionally found.

should seal it in his presence, calling his attention to the fact. Before leaving the premises, place upon the bottle a label or tag, with the date, the name of the owner, and the name of the officer upon it, and also the name of the town or city. Then place in a box, with the certificate required by law, and forward without delay to the analyst.

FORM OF LABEL.

Town .....
Date of seizure 19
Owner. .....
Kind of liquor. .....
Brewer .....

Accompanying each sample is a certificate like the following, the first part of which is filled out and signed by the officer, while the second part, containing the data of analysis, is filled out and signed by the analyst and returned by him to the officer. Such a certificate is nearly always accepted as evidence in court without the personal appearance of the analyst.

.....ss .....19 .

To the State Board of Health:

I send herewith a sample of.....
taken from liquors seized by me.....19 .

Ascertain the percentage of alcohol it contains, by volume, at sixty degrees Fahrenheit, and return to me a certificate herewith upon the annexed form.

Seized from.....

.....
.....
Officer.



A convenient method for recording analyses is by the employment of numbered library cards, which bear the same number as the certificates and are kept by the analyst.

The following is a convenient form:

No. .....
County .....
City or town. .....
Officer. .....
Defendant. .....
Address. .....
Kind of liquor. .....
Seized. .....
Received. .....
How delivered .....
Sealed. .....
Condition. .....
Kind of bottle. .....
Registered. .....

Analyzed. .....
Wt. flask and alc .....
Wt. flask. .....
Wt. alc. .....
Sp. gr. alc. (60°). .....
Per cent alcohol. .....
Reported. .....

METHODS OF ANALYSIS COMMON TO ALL LIQUORS.

Specific Gravity.—This should be taken at 15.6° or calculated to that temperature. The most convenient mode of procedure is to bring the temperature of the sample somewhat below that point by allowing the flask containing it to stand in cold water, and to have everything in readiness to make the determination when 15.6° temperature has been reached, either by the hydrometer spindle in a glass cylinder, by the Westphal balance, or by the pycnometer. The latter is by far the most accurate, especially if it is of the form which is fitted with a thermometer-stopper.

Detection of Alcohol.—It is rarely necessary to make a qualitative test for alcohol in liquors, since it is almost invariably present even in many of the so-called temperance drinks, at least in small amount. Indeed in many localities a beverage is legally a temperance drink that contains not more than 1% alcohol by volume.

The Iodoform Test.—Alcohol, when present in aqueous solution to the extent of 0.1% or more, may be detected by the iodoform test. The solution is warmed in a test-tube with a few drops of a strong solution of iodine in potassium iodide, after which enough sodium hydroxide solution is added to nearly decolorize. On standing for some time a yellow precipitate of iodoform will appear if alcohol be present, or at once if there is a considerable amount, and the characteristic odor of iodoform will be rendered apparent, even when the precipitate is so slight as to be almost imperceptible. This iodoform precipitate is crystalline, showing under the microscope as star-shaped groups or hexagonal tablets.

It should not be forgotten that other substances than alcohol give the reaction, as lactic acid, acetone, and various aldehydes and ketones.

Pure methyl or amyl alcohol or acetic acid do not thus react.

Berthelot recommends benzoyl chloride as a reagent for detecting alcohol. By warming a mixture of a few drops of benzoyl chloride with the solution to be tested, and adding a little sodium hydroxide, ethyl benzoate is formed, recognizable by its distinctive odor. This reaction is delicate to 0.1% alcohol. The presence of other alcohols than ethyl produces ethers of characteristic odor.

Hardy's Test for Alcohol consists in shaking the aqueous solution with some powdered guaiacum resin, filtering, and adding to the filtrate a little hydrocyanic acid and a drop of dilute copper sulphate solution. A blue coloration considerably deeper than that due to the copper salt is indicative of alcohol.

Methyl Alcohol in spirits is tested for by Mulliken's reaction (p. 743).

DETERMINATION OF ALCOHOL.—In the case of carbonated liquids it is necessary to first expel the free carbon dioxide, which is readily accomplished by pouring the liquor back and forth from one beaker to another, from time to time removing the excess of froth from the top of the vessel by the aid of the hand. Or, the sample may be shaken vigorously in a large separatory funnel, and the still liquor drawn off from below the froth, repeating the operation several times if necessary. In either case the mechanical treatment should be continued till the liquor is comparatively quiet and free from foam.

If the liquor is perceptibly acid, the acidity should first be neutralized with sodium hydroxide or other alkali, to guard against the passing over of volatile acid. This is usually unnecessary with beer or distilled liquors, wherein the volatile acid is not generally present in sufficient quantity

for convenience and accuracy in the original flask in which it was measured. Nearly all alcoholic liquors, if comparatively free from carbon dioxide, will boil without undue frothing or foaming. New wine will occasionally give trouble in this regard, but foaming may usually be prevented in this case by the addition of tannic acid. In case of wine, cider, and beer all the alcohol will have passed over in the first 75 cc. of the distillate, or three-fourths the original measured volume, but with distilled liquors high in alcohol the process had better be continued till nearly 100 cc. or the original volume taken have passed over. If the condenser is of glass, one can observe when all the alcohol has been distilled over, for the reason that the mixed alcohol and water vapors in the upper portion of the condenser present a striated or wavy appearance, readily apparent so long as the alcohol is passing over, while after all the alcohol has been distilled, the condenser-tube appears perfectly clear. The distillation is thus continued for some time after this striated appearance has ceased. The distillate in the receiving glass is finally made up to the mark or to the original volume of the liquor taken. Strictly speaking, the measurements before and after distillation should be made at 15.6° C., but, excepting in case of distilled liquors, no appreciable error results from making both measurements at the same or room temperature. Another precaution formerly thought necessary was to have the delivery-tube from the condenser pass below the level of a little water in the receiving-flask from the start, but equally accurate results have been obtained by simply allowing the end of the condenser-tube to enter the narrow-necked flask.

Fig. 111 shows a bank of six stills of the kind used in the author's laboratory for alcohol determination in liquors. In each still the vertical glass worm-condenser, the round-bottomed distilling-flask, and the lamp, are supported by rings held by a single upright rod. The receiving-flask is readily connected with the condenser by means of a single bent tube provided with a rubber stopper. The cold-water pipe supplying the condensers is shown at the top, and the gas-supply pipe at the bottom.

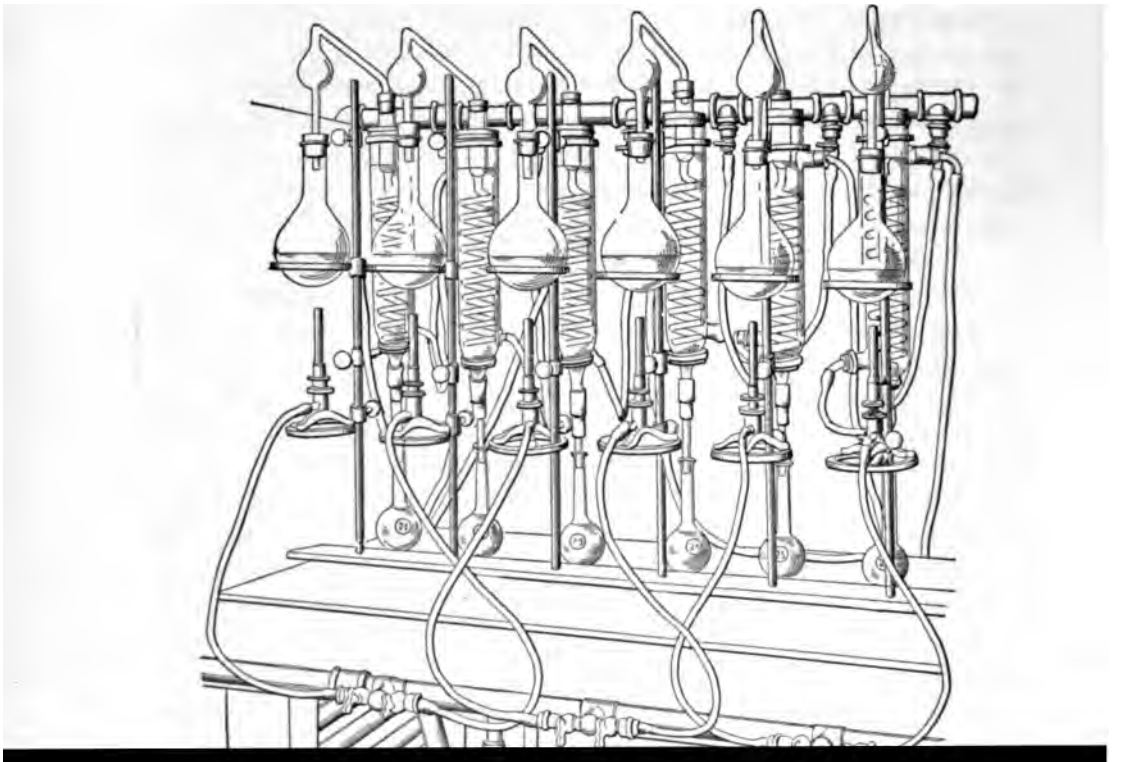
The distillate, made up to the original volume, is thoroughly shaken and its specific gravity taken at exactly 15.6° in a pycnometer, or by the Westphal balance. From the specific gravity the corresponding percentage of alcohol by volume is ascertained by reference to the accompanying tables.

If alcohol by weight is desired, operate on 100 grams of the sample, making up the distillate to the same weight.

(2) **From the Specific Gravity of the Sample.**—In the case of distilled liquors having very little residue, an approximation to the true

percentage of alcohol may be obtained by using the alcohol table in connection with the specific gravity of the liquor itself. The accuracy of this method depends largely on the freedom from residue, being absolutely correct for mixtures of alcohol and water only.

(3) **By Evaporation.**—Determine the specific gravity of the sample, evaporate a measured portion of the liquor (50 or 100 cc.) in a porcelain



SPECIFIC GRAVITY AND PERCENTAGE OF ALCOHOL.

(ACCORDING TO HEHNER.)

Spec. Grav. at 15.6° C.	Absolute Alcohol. Per Cent by Weight	Per Cent by Volume.	Grams per 100 cc.	Spec. Grav. at 15.6° C.	Absolute Alcohol. Per Cent by Weight	Per Cent by Volume.	Grams per 100 cc.	Spec. Grav. at 15.6° C.	Absolute Alcohol. Per Cent by Weight	Per Cent by Volume.	Grams per 100 cc.
1.0000	0.00	0.00	0.00								
0.9999	0.05	0.07	0.05	0.9959	2.33	2.93	2.32	0.9919	4.69	5.86	4.65
8	0.11	0.13	0.11	8	2.39	3.00	2.38	8	4.75	5.94	4.71
7	0.16	0.20	0.16	7	2.44	3.07	2.43	7	4.81	6.02	4.77
6	0.21	0.26	0.21	6	2.50	3.14	2.49	6	4.87	6.10	4.83
5	0.26	0.33	0.26	5	2.56	3.21	2.55	5	4.94	6.17	4.90
4	0.32	0.40	0.32	4	2.61	3.28	2.60	4	5.00	6.24	4.95
3	0.37	0.46	0.37	3	2.67	3.35	2.65	3	5.06	6.32	5.01
2	0.42	0.53	0.42	2	2.72	3.42	2.70	2	5.12	6.40	5.07
1	0.47	0.60	0.47	1	2.78	3.49	2.76	1	5.19	6.48	5.14
0	0.53	0.66	0.53	0	2.83	3.55	2.81	0	5.25	6.55	5.20
0.9989	0.58	0.73	0.58	0.9949	2.89	3.62	2.87	0.9909	5.31	6.63	5.26
8	0.63	0.79	0.63	8	2.94	3.69	2.92	8	5.37	6.71	5.32
7	0.68	0.86	0.68	7	3.00	3.76	2.98	7	5.44	6.78	5.39
6	0.74	0.93	0.74	6	3.06	3.83	3.04	6	5.50	6.86	5.45
5	0.79	0.99	0.79	5	3.12	3.90	3.10	5	5.56	6.94	5.51
4	0.84	1.06	0.84	4	3.18	3.98	3.16	4	5.62	7.01	5.57
3	0.89	1.13	0.89	3	3.24	4.05	3.22	3	5.69	7.09	5.64
2	0.95	1.19	0.95	2	3.29	4.12	3.27	2	5.75	7.17	5.70
1	1.00	1.26	1.00	1	3.35	4.20	3.33	1	5.81	7.25	5.76
0	1.06	1.34	1.06	0	3.41	4.27	3.39	0	5.87	7.32	5.81
0.9979	1.12	1.42	1.12	0.9939	3.47	4.34	3.45	0.9899	5.94	7.40	5.88
8	1.19	1.49	1.19	8	3.53	4.42	3.51	8	6.00	7.48	5.94
7	1.25	1.57	1.25	7	3.59	4.49	3.57	7	6.07	7.57	6.01
6	1.31	1.65	1.31	6	3.65	4.56	3.63	6	6.14	7.66	6.07
5	1.37	1.73	1.37	5	3.71	4.63	3.69	5	6.21	7.74	6.14
4	1.44	1.81	1.44	4	3.76	4.71	3.74	4	6.28	7.83	6.21
3	1.50	1.88	1.50	3	3.82	4.78	3.80	3	6.36	7.92	6.29
2	1.56	1.96	1.56	2	3.88	4.85	3.85	2	6.43	8.01	6.36
1	1.62	2.04	1.61	1	3.94	4.93	3.91	1	6.50	8.10	6.43
0	1.69	2.12	1.68	0	4.00	5.00	3.97	0	6.57	8.18	6.50
0.9969	1.75	2.20	1.74	0.9929	4.06	5.08	4.03	0.9889	6.64	8.27	6.57
8	1.81	2.27	1.80	8	4.12	5.16	4.09	8	6.71	8.36	6.63
7	1.87	2.35	1.86	7	4.19	5.24	4.16	7	6.78	8.45	6.70
6	1.94	2.43	1.93	6	4.25	5.32	4.22	6	6.86	8.54	6.78
5	2.00	2.51	1.99	5	4.31	5.39	4.28	5	6.93	8.63	6.85
4	2.06	2.58	2.05	4	4.37	5.47	4.34	4	7.00	8.72	6.92
3	2.11	2.62	2.10	3	4.44	5.55	4.40	3	7.07	8.80	6.99
2	2.17	2.72	2.16	2	4.50	5.63	4.46	2	7.13	8.88	7.05
1	2.22	2.79	2.21	1	4.56	5.71	4.52	1	7 20	8.96	7.12
0	2.28	2.86	2.27	0	4.62	5.78	4.58	0	7.27	9.04	7.19

SPECIFIC GRAVITY AND PERCENTAGE OF ALCOHOL—(*Continued*).

Spec. Grav. at 15.6° C.	Absolute Alcohol. Per Cent by Weight	Per Cent by Volume.	Grams per 100 cc.	Spec. Grav. at 15.6° C.	Absolute Alcohol. Per Cent by Weight	Per Cent by Volume.	Grams per 100 cc.	Spec. Grav. at 15.6° C.	Absolute Alcohol. Per Cent by Weight	Per Cent by Volume.	Grams per 100 cc.
0.9879	7.33	9.13	7.24	0.9829	10.92	13.52	10.73	0.9779	14.91	18.36	14.58
8	7.40	9.21	7.31	8	11.00	13.62	10.81	8	15.00	18.48	14.66
7	7.47	9.29	7.37	7	11.08	13.71	10.89	7	15.08	18.58	14.74
6	7.53	9.37	7.43	6	11.15	13.81	10.95	6	15.17	18.68	14.83
5	7.60	9.45	7.50	5	11.23	13.90	11.03	5	15.25	18.78	14.90
4	7.67	9.54	7.57	4	11.31	13.99	11.11	4	15.33	18.88	14.98
3	7.73	9.62	7.63	3	11.38	14.09	11.18	3	15.42	18.98	15.07
2	7.80	9.70	7.70	2	11.46	14.18	11.26	2	15.50	19.08	15.14
1	7.87	9.78	7.77	1	11.54	14.27	11.33	1	15.58	19.18	15.21
0	7.93	9.86	7.83	0	11.62	14.37	11.41	0	15.67	19.28	15.30
0.9869	8.00	9.95	7.89	0.9819	11.69	14.46	11.48	0.9769	15.75	19.39	15.38
8	8.07	10.03	7.96	8	11.77	14.56	11.56	8	15.83	19.49	15.46
7	8.14	10.12	8.04	7	11.85	14.65	11.64	7	15.92	19.59	15.54
6	8.21	10.21	8.10	6	11.92	14.74	11.70	6	16.00	19.68	15.62
5	8.29	10.30	8.17	5	12.00	14.84	11.78	5	16.08	19.78	15.70
4	8.36	10.38	8.24	4	12.08	14.93	11.85	4	16.15	19.87	15.76
3	8.43	10.47	8.31	3	12.15	15.02	11.92	3	16.23	19.96	15.84
2	8.50	10.56	8.38	2	12.23	15.12	12.00	2	16.31	20.06	15.90
1	8.57	10.65	8.45	1	12.31	15.21	12.08	1	16.38	20.15	15.99
0	8.64	10.73	8.52	0	12.38	15.30	12.14	0	16.46	20.24	16.06
0.9859	8.71	10.82	8.58	0.9809	12.46	15.40	12.22	0.9759	16.54	20.33	16.13
8	8.79	10.91	8.66	8	12.54	15.49	12.30	8	16.62	20.43	16.21
7	8.86	11.00	8.73	7	12.62	15.58	12.37	7	16.69	20.52	16.28
6	8.93	11.08	8.80	6	12.69	15.68	12.44	6	16.77	20.61	16.35
5	9.00	11.17	8.87	5	12.77	15.77	12.51	5	16.85	20.71	16.43
4	9.07	11.26	8.93	4	12.85	15.86	12.59	4	16.92	20.80	16.50
3	9.14	11.35	9.00	3	12.92	15.96	12.66	3	17.00	20.89	16.57
2	9.21	11.44	9.07	2	13.00	16.05	12.74	2	17.08	20.99	16.65
1	9.29	11.52	9.14	1	13.08	16.15	12.81	1	17.17	21.09	16.74
0	9.36	11.61	9.22	0	13.15	16.24	12.89	0	17.25	21.19	16.81
0.9849	9.43	11.70	9.29	0.9799	13.23	16.33	12.96	0.9749	17.33	21.29	16.89
8	9.50	11.79	9.35	8	13.31	16.43	13.03	8	17.42	21.39	16.97

SPECIFIC GRAVITY AND PERCENTAGE OF ALCOHOL—(*Continued*).

Spec. Grav. at 15.6° C.	Absolute Alcohol. Per Cent by Weight	Per Cent by Volume.	Grams per 100 cc.	Spec. Grav. at 15.6° C.	Absolute Alcohol. Per Cent by Weight	Per Cent by Volume.	Grams per 100 cc.	Spec. Grav. at 15.6° C.	Absolute Alcohol. Per Cent by Weight	Per Cent by Volume.	Grams per 100 cc.
0.9729	18.92	23.19	18.41	0.9679	22.92	27.95	22.18	0.9629	26.60	32.27	25.61
8	19.00	23.18	18.48	8	23.00	28.04	22.26	8	26.67	32.34	25.67
7	19.08	23.38	18.56	7	23.08	28.13	22.33	7	26.73	32.42	25.73
6	19.17	23.48	18.65	6	23.15	28.22	22.40	6	26.80	32.50	25.79
5	19.25	23.58	18.73	5	23.23	28.31	22.47	5	26.87	32.58	25.85
4	19.33	23.68	18.80	4	23.31	28.41	22.54	4	26.93	32.65	25.91
3	19.42	23.78	18.88	3	23.38	28.50	22.61	3	27.00	32.73	25.98
2	19.50	23.88	18.95	2	23.46	28.59	22.69	2	27.07	32.81	26.04
1	19.58	23.98	19.03	1	23.54	28.68	22.76	1	27.14	32.90	26.10
0	19.67	24.08	19.12	0	23.62	28.77	22.83	0	27.21	32.98	26.17
0.9719	19.75	24.18	19.19	0.9669	23.69	28.86	22.90	0.9619	27.29	33.06	26.25
8	19.83	24.28	19.27	8	23.77	28.95	22.97	8	27.36	33.15	26.31
7	19.92	24.38	19.36	7	23.85	29.04	23.05	7	27.43	33.23	26.37
6	20.00	24.48	19.44	6	23.92	29.13	23.11	6	27.50	33.31	26.43
5	20.08	24.58	19.51	5	24.00	29.22	23.19	5	27.57	33.39	26.51
4	20.17	24.68	19.59	4	24.08	29.31	23.27	4	27.64	33.48	26.57
3	20.25	24.78	19.66	3	24.15	29.40	23.33	3	27.71	33.56	26.64
2	20.33	24.88	19.74	2	24.23	29.49	23.40	2	27.79	33.64	26.71
1	20.42	24.98	19.83	1	24.31	29.58	23.48	1	27.86	33.73	26.78
0	20.50	25.07	19.90	0	24.38	29.67	23.55	0	27.93	33.81	26.84
0.9709	20.58	25.17	19.98	0.9659	24.46	29.76	23.62	0.9609	28.00	33.89	26.90
8	20.67	25.27	20.07	8	24.54	29.86	23.70	8	28.06	33.97	26.96
7	20.75	25.37	20.14	7	24.62	29.95	23.77	7	28.12	34.04	27.01
6	20.83	25.47	20.22	6	24.69	30.04	23.84	6	28.19	34.11	27.07
5	20.92	25.57	20.30	5	24.77	30.13	23.91	5	28.25	34.18	27.13
4	21.00	25.67	20.33	4	24.85	30.22	23.99	4	28.31	34.25	27.18
3	21.08	25.76	20.46	3	24.92	30.31	24.05	3	28.37	34.33	27.24
2	21.15	25.86	20.52	2	25.00	30.40	24.12	2	28.44	34.40	27.31
1	21.23	25.95	20.59	1	25.07	30.48	24.19	1	28.50	34.47	27.36
0	21.31	26.04	20.67	0	25.14	30.57	24.26	0	28.56	34.54	27.42
0.9699	21.38	26.13	20.73	0.9649	25.21	30.65	24.32	0.9599	28.62	34.61	27.47
8	21.46	26.22	20.81	8	25.29	30.73	24.39	8	28.69	34.69	27.53
7	21.54	26.31	20.89	7	25.36	30.82	24.46	7	28.75	34.76	27.59
6	21.62	26.40	20.96	6	25.43	30.90	24.53	6	28.81	34.83	27.64
5	21.69	26.49	21.03	5	25.50	30.98	24.59	5	28.87	34.90	27.70
4	21.77	26.58	21.11	4	25.57	31.07	24.66	4	28.94	34.97	27.76
3	21.85	26.67	21.18	3	25.64	31.13	24.72	3	29.00	35.05	27.82
2	21.92	26.77	21.25	2	25.71	31.23	24.79	2	29.07	35.12	27.89
1	22.00	26.86	21.33	1	25.79	31.32	24.86	1	29.13	35.20	27.95
0	22.08	26.95	21.40	0	25.86	31.40	24.93	0	29.20	35.28	28.00
0.9689	22.15	27.04	21.47	0.9639	25.93	31.48	24.99	0.9589	29.27	35.35	28.07
8	22.23	27.13	21.54	8	26.00	31.57	25.06	8	29.33	35.43	28.12
7	22.31	27.22	21.61	7	26.07	31.65	25.12	7	29.40	35.51	28.18
6	22.38	27.31	21.68	6	26.13	31.72	25.18	6	29.47	35.58	28.24
5	22.46	27.40	21.76	5	26.20	31.80	25.23	5	29.53	35.66	28.30
4	22.54	27.49	21.83	4	26.27	31.88	25.30	4	29.60	35.74	28.36
3	22.62	27.59	21.90	3	26.33	31.96	25.36	3	29.67	35.81	28.43
2	22.69	27.68	21.96	2	26.40	32.03	25.43	2	29.73	35.89	28.48
1	22.77	27.77	22.01	1	26.47	32.11	25.49	1	29.80	35.97	28.54
0	22.85	27.86	22.12	0	26.53	32.19	25.55	0	29.87	36.04	28.61

SPECIFIC GRAVITY AND PERCENTAGE OF ALCOHOL—(*Continued*).

Spec. Grav. at 15.6° C.	Absolute Alcohol. Per Cent by Weight	Per Cent by Volume.	Grams per 100 cc.	Spec. Grav. at 15.6° C.	Absolute Alcohol. Per Cent by Weight	Per Cent by Volume.	Grams per 100 cc.	Spec. Grav. at 15.6° C.	Absolute Alcohol. Per Cent by Weight	Per Cent by Volume.	Grams per 100 cc.
0.9579	29.93	36.12	28.67	0.9529	32.94	39.54	31.38	0.9479	35.55	42.45	33.70
8	30.00	36.20	28.73	8	33.00	39.61	31.43	8	35.60	42.51	33.75
7	30.06	36.26	28.78	7	33.06	39.68	31.48	7	35.65	42.56	33.79
6	30.11	36.32	28.82	6	33.12	39.74	31.53	6	35.70	42.62	33.83
5	30.17	36.39	28.88	5	33.18	39.81	31.59	5	35.75	42.67	33.88
4	30.22	36.45	28.92	4	33.24	39.87	31.63	4	35.80	42.73	33.92
3	30.28	36.51	28.98	3	33.29	39.94	31.69	3	35.85	42.78	33.97
2	30.33	36.57	29.03	2	33.35	40.01	31.74	2	35.90	42.84	34.01
1	30.39	36.64	29.08	1	33.41	40.07	31.80	1	35.95	42.89	34.05
0	30.44	36.70	29.13	0	33.47	40.14	31.86	0	36.00	42.95	34.09
0.9569	30.50	36.76	29.18	0.9519	33.53	40.20	31.91	0.9469	36.06	43.01	34.14
8	30.56	36.83	29.23	8	33.59	40.27	31.96	8	36.11	43.07	34.09
7	30.61	36.89	29.27	7	33.65	40.34	32.01	7	36.17	43.13	34.24
6	30.67	36.95	29.33	6	33.71	40.40	32.07	6	36.22	43.19	34.28
5	30.72	37.02	29.38	5	33.76	40.47	32.12	5	36.28	43.26	34.34
4	30.78	37.08	29.43	4	33.82	40.53	32.17	4	36.33	43.32	34.38
3	30.83	37.14	29.48	3	33.88	40.60	32.22	3	36.39	43.38	34.44
2	30.89	37.20	29.53	2	33.94	40.67	32.27	2	36.44	43.44	34.48
1	30.94	37.27	29.58	1	34.00	40.74	32.32	1	36.50	43.50	34.54
0	31.00	37.34	29.63	0	34.05	40.79	32.37	0	36.56	43.56	34.58
0.9559	31.06	37.41	29.69	0.9509	34.10	40.84	32.41	0.9459	36.61	43.63	34.63
8	31.12	37.48	29.74	8	34.14	40.90	32.45	8	36.67	43.69	34.69
7	31.19	37.55	29.81	7	34.19	40.95	32.49	7	36.72	43.75	34.73
6	31.25	37.62	29.86	6	34.24	41.00	32.54	6	36.78	43.81	34.79
5	31.31	37.69	29.91	5	34.29	41.05	32.59	5	36.83	43.87	34.83
4	31.37	37.76	29.97	4	34.33	41.11	32.63	4	36.89	43.93	34.88
3	31.44	37.83	30.03	3	34.38	41.16	32.67	3	36.94	44.00	34.92
2	31.50	37.90	30.09	2	34.43	41.21	32.71	2	37.00	44.06	34.96
1	31.56	37.97	30.14	1	34.48	41.26	32.75	1	37.06	44.12	35.02
0	31.62	38.04	30.20	0	34.52	41.32	32.79	0	37.11	44.18	35.07
0.9549	31.69	38.11	30.26	0.9499	34.57	41.37	32.84	0.9449	37.17	44.24	35.12
8	31.75	38.18	30.31	8	34.62	41.42	32.88	8	37.22	44.30	35.16
7	31.81	38.25	30.36	7	34.67	41.48	32.92	7	37.28	44.36	35.21



SPECIFIC GRAVITY AND PERCENTAGE OF ALCOHOL—(*Continued*).

Spec. Grav. at 15.6° C.	Absolute Alcohol. Per Cent by Weight	Per Cent by Volume.	Grams per 100 cc.	Spec. Grav. at 15.6° C.	Absolute Alcohol. Per Cent by Weight	Per Cent by Volume.	Grams per 100 cc.	Spec. Grav. at 15.6° C.	Absolute Alcohol. Per Cent by Weight	Per Cent by Volume.	Grams per 100 cc.
0.9420	38.28	45.47	36.08	0.9379	40.85	48.26	38.31	0.9329	43.29	50.87	40.38
8	38.33	45.53	36.13	8	40.90	48.32	38.35	8	43.33	50.92	40.42
7	38.39	45.59	36.18	7	40.95	48.37	38.39	7	43.39	50.97	40.46
6	38.44	45.65	36.23	6	41.00	48.43	38.44	6	43.43	51.02	40.50
5	38.50	45.71	36.28	5	41.05	48.48	38.48	5	43.48	51.07	40.54
4	38.56	45.77	36.33	4	41.10	48.54	38.52	4	43.52	51.12	40.58
3	38.61	45.83	36.38	3	41.15	48.59	38.58	3	43.57	51.17	40.62
2	38.67	45.89	36.43	2	41.20	48.64	38.62	2	43.62	51.22	40.66
1	38.72	45.95	36.48	1	41.25	48.70	38.66	1	43.67	51.27	40.70
0	38.78	46.02	36.53	0	41.30	48.75	38.70	0	43.71	51.32	40.74
0.9419	38.83	46.08	36.57	0.9369	41.35	48.80	38.74	0.9319	43.76	51.38	40.78
8	38.89	46.14	36.62	8	41.40	48.86	38.78	8	43.81	51.43	40.81
7	38.94	46.20	36.67	7	41.45	48.91	38.82	7	43.86	51.48	40.85
6	39.00	46.26	36.72	6	41.50	48.97	38.87	6	43.90	51.53	40.89
5	39.05	46.32	36.76	5	41.55	49.02	38.91	5	43.95	51.58	40.93
4	39.10	46.37	36.80	4	41.60	49.07	38.95	4	44.00	51.63	40.97
3	39.15	46.42	36.85	3	41.65	49.13	38.99	3	44.05	51.68	41.01
2	39.20	46.48	36.89	2	41.70	49.18	39.04	2	44.09	51.72	41.05
1	39.25	46.53	36.94	1	41.75	49.23	39.08	1	44.14	51.77	41.09
0	39.30	46.59	36.98	0	41.80	49.29	39.13	0	44.18	51.82	41 13
0 9409	39.35	46.64	37.02	0.9359	41.85	49.34	39.17	0.9309	44.23	51.87	41.17
8	39.40	46.70	37.07	8	41.90	49.40	39.21	8	44.27	51.91	41.20
7	39.45	46.75	37.11	7	41.95	49.45	39.25	7	44.32	51.96	41.24
6	39.50	46.80	37.15	6	42.00	49 50	39.30	6	44.36	52.01	41.28
5	39.55	46.86	37.19	5	42.05	49.55	39.34	5	44.41	52.06	41.31
4	39.60	46.91	37.23	4	42.10	49.61	39.38	4	44.46	52.10	41.35
3	39.65	46.97	37.27	3	42.14	49.66	39.42	3	44.50	52.15	41.49
2	39.70	47.02	37.32	2	42.19	49.71	39.46	2	44.55	52.20	41.43
1	39.75	47.08	37.36	1	42.24	49.76	39.50	1	44.59	52.25	41.47
0	39.80	47.13	37.41	0	42.29	49.81	39.54	0	44.64	52.29	41.51
0.9399	39.85	47.18	37.45	0.9349	42.33	49.86	39.58	0.9299	44.68	52.34	41.55
8	39.90	47.24	37.49	8	42.38	49.91	39.62	8	44.73	52.39	41.59
7	39.95	47.29	37.53	7	42.43	49.96	39.66	7	44.77	52.44	41.63
6	40.00	47.35	37.58	6	42.48	50.01	39.70	6	44.82	52.48	41.67
5	40.05	47.40	37.62	5	42.52	50.06	39.74	5	44.86	52.53	41.70
4	40.10	47.45	37.67	4	42.57	50.11	39.78	4	44.91	52.58	41.74
3	40.15	47.51	37.71	3	42.62	50.16	39.82	3	44.96	52.63	41.77
2	40.20	47.56	37.75	2	42.67	50.21	39.86	2	45.00	52.68	41.81
1	40.25	47.62	37.80	1	42.71	50.26	39.90	1	45.05	52.72	41.85
0	40.30	47.67	37.84	0	42.76	50.31	39.94	0	45.09	52.77	41.89
0.9389	40.35	47.72	37.88	0.9339	42.81	50.37	39.98	0.9289	45.14	52.82	41.93
8	40.40	47.78	37.92	8	42.86	50.42	40.02	8	45.18	52.87	41.97
7	40.45	47.83	37.96	7	42.90	50.47	40.06	7	45.23	52.91	42.00
6	40.50	47.89	38.00	6	42.95	50.52	40.10	6	45.27	52.96	42.04
5	40.55	47.94	38.05	5	43.00	50.57	40.14	5	45.32	53.01	42.08
4	40.60	47.99	38.09	4	43.05	50.62	40.18	4	45.36	53.06	42.12
3	40.65	48.05	38.13	3	43.10	50.67	40.22	3	45.41	53.10	42.16
2	40.70	48.10	38.18	2	43.13	50.72	40.26	2	45.46	53.15	42.19
1	40.75	48.16	38.22	1	43.19	50.77	40.30	1	45.50	53.20	42.23
0	40.80	48.21	38.27	0	43.24	50.82	40.34	0	45.55	53.24	42.27

SPECIFIC GRAVITY AND PERCENTAGE OF ALCOHOL—(*Continued*).

Spec. Grav. at 15.6° C.	Absolute Alcohol.		Spec. Grav. at 15.6° C.	Absolute Alcohol.		Spec. Grav. at 15.6° C.	Absolute Alcohol.	
	Per Cent by Weight.	Per Cent by Volume.		Per Cent by Weight.	Per Cent by Volume.		Per Cent by Weight.	Per Cent by Volume.
0.9279	45.59	53.29	0.9229	47.86	55.65	0.9179	50.13	57.97
8	45.64	53.34	8	47.91	55.69	8	50.17	58.01
7	45.68	53.39	7	47.96	55.74	7	50.22	58.06
6	45.73	53.43	6	48.00	55.79	6	50.26	58.10
5	45.77	53.48	5	48.05	55.83	5	50.30	58.14
4	45.82	53.53	4	48.09	55.88	4	50.35	58.19
3	45.86	53.58	3	48.14	55.93	3	50.39	58.23
2	45.91	53.62	2	48.18	55.97	2	50.43	58.28
1	45.96	53.67	1	48.23	56.02	1	50.48	58.32
0	46.00	53.72	0	48.27	56.07	0	50.52	58.36
0.9269	46.05	53.77	0.9219	48.32	56.11	0.9169	50.57	58.41
8	46.09	53.81	8	48.36	56.16	8	50.61	58.45
7	46.14	53.86	7	48.41	56.21	7	50.65	58.50
6	46.18	53.91	6	48.46	56.25	6	50.70	58.54
5	46.23	53.95	5	48.50	56.30	5	50.74	58.58
4	46.27	54.00	4	48.55	56.35	4	50.78	58.63
3	46.32	54.05	3	48.59	56.40	3	50.83	58.67
2	46.36	54.10	2	48.64	56.44	2	50.87	58.72
1	46.41	54.14	1	48.68	56.49	1	50.91	58.76
0	46.46	54.19	0	48.73	56.54	0	50.96	58.80
0.9259	46.50	54.24	0.9209	48.77	56.58	0.9159	51.00	58.85
8	46.55	54.29	8	48.82	56.63	8	51.04	58.89
7	46.59	54.33	7	48.86	56.68	7	51.08	58.93
6	46.64	54.38	6	48.91	56.72	6	51.13	58.97
5	46.68	54.43	5	48.96	56.77	5	51.17	59.01
4	46.73	54.47	4	49.00	56.82	4	51.21	59.05
3	46.77	54.52	3	49.04	56.86	3	51.25	59.09
2	46.82	54.57	2	49.08	56.90	2	51.29	59.14
1	46.86	54.62	1	49.12	56.94	1	51.33	59.18
0	46.91	54.66	0	49.16	56.98	0	51.38	59.22
0.9249	46.96	54.71	0.9199	49.20	57.02	0.9149	51.42	59.26
8	47.00	54.76	Proof 8	49.24	57.06	8	51.46	59.30
7	47.05	54.80	7	49.29	57.10	7	51.50	59.34

SPECIFIC GRAVITY AND PERCENTAGE OF ALCOHOL—(*Continued*).

Spec. Grav. at 15.6° C.	Absolute Alcohol.		Spec. Grav. at 15.6° C.	Absolute Alcohol.		Spec. Grav. at 15.6° C.	Absolute Alcohol.	
	Per Cent by Weight.	Per Cent by Volume.		Per Cent by Weight.	Per Cent by Volume.		Per Cent by Weight.	Per Cent by Volume.
0.9129	52.27	60.12	0.9079	54.52	62.36	0.9029	56.82	64.63
8	52.32	60.16	8	54.57	62.41	8	56.86	64.67
7	52.36	60.21	7	54.62	62.45	7	56.91	64.71
6	52.41	60.25	6	54.67	62.50	6	56.95	64.76
5	52.45	60.30	5	54.71	62.55	5	57.00	64.80
4	52.50	60.34	4	54.76	62.60	4	57.04	64.85
3	52.55	60.39	3	54.81	62.65	3	57.08	64.89
2	52.59	60.44	2	54.86	62.69	2	57.13	64.93
1	52.64	60.47	1	54.90	62.74	1	57.17	64.97
0	52.68	60.52	0	54.95	62.79	0	57.21	65.01
0.9119	52.73	60.56	0.9069	55.00	62.84	0.9019	57.25	65.05
8	52.77	60.61	8	55.05	62.88	8	57.29	65.09
7	52.82	60.65	7	55.09	62.93	7	57.33	65.13
6	52.86	60.70	6	55.14	62.97	6	57.38	65.17
5	52.91	60.74	5	55.18	63.02	5	57.42	65.21
4	52.95	60.79	4	55.23	63.06	4	57.46	65.25
3	53.00	60.85	3	55.27	63.11	3	57.50	65.29
2	53.04	60.89	2	55.32	63.15	2	57.54	65.33
1	53.09	60.93	1	55.36	63.20	1	57.58	65.37
0	53.13	60.97	0	55.41	63.24	0	57.63	65.41
0.9109	53.17	61.02	0.9059	55.45	63.28	0.9009	57.67	65.45
8	53.22	61.06	8	55.50	63.33	8	57.71	65.49
7	53.26	61.10	7	55.55	63.37	7	57.75	65.53
6	53.30	61.15	6	55.59	63.42	6	57.79	65.57
5	53.35	61.19	5	55.64	63.46	5	57.83	65.61
4	53.39	61.23	4	55.68	63.51	4	57.88	65.65
3	53.43	61.28	3	55.73	63.55	3	57.92	65.69
2	53.48	61.32	2	55.77	63.60	2	57.96	65.73
1	53.52	61.36	1	55.82	63.64	1	58.00	65.77
0	53.57	61.40	0	55.86	63.69	0	58.05	65.81
0.9099	53.61	61.45	0.9049	55.91	63.73	0.8999	58.09	65.85
8	53.65	61.49	8	55.95	63.78	8	58.14	65.90
7	53.70	61.53	7	56.00	63.82	7	58.18	65.94
6	53.74	61.58	6	56.05	63.87	6	58.23	65.99
5	53.78	61.62	5	56.09	63.91	5	58.27	66.03
4	53.83	61.66	4	56.14	63.96	4	58.32	66.07
3	53.87	61.71	3	56.18	64.00	3	58.36	66.12
2	53.91	61.75	2	56.23	64.05	2	58.41	66.16
1	53.96	61.79	1	56.27	64.09	1	58.45	66.21
0	54.00	61.84	0	56.32	64.14	0	58.50	66.25
0.9089	54.05	61.88	0.9039	56.36	64.18	0.8989	58.55	66.29
8	54.10	61.93	8	56.41	64.22	8	58.59	66.34
7	54.14	61.98	7	56.45	64.27	7	58.64	66.38
6	54.19	62.03	6	56.30	64.31	6	58.68	66.43
5	54.24	62.07	5	56.55	64.36	5	58.73	66.47
4	54.29	62.12	4	56.59	64.40	4	58.77	66.51
3	54.33	62.17	3	56.64	64.45	3	58.82	66.56
2	54.38	62.22	2	56.68	64.49	2	58.86	66.60
1	54.43	62.26	1	56.73	64.54	1	58.91	66.65
0	54.48	62.31	0	56.77	64.58	0	58.95	66.69

SPECIFIC GRAVITY AND PERCENTAGE OF ALCOHOL—(*Continued*).

Spec. Grav. at 15.6° C.	Absolute Alcohol.		Spec. Grav. at 15.6° C.	Absolute Alcohol.		Spec. Grav. at 15.6° C.	Absolute Alcohol.	
	Per Cent by Weight.	Per Cent by Volume.		Per Cent by Weight.	Per Cent by Volume.		Per Cent by Weight.	Per Cent by Volume.
0.8979	59.00	66.74	0.8929	61.13	68.76	0.8879	63.30	70.81
8	59.04	66.78	8	61.17	68.80	8	63.35	70.85
7	59.09	66.82	7	61.21	68.83	7	63.39	70.89
6	59.13	66.86	6	61.25	68.87	6	63.43	70.93
5	59.17	66.90	5	61.29	68.91	5	63.48	70.97
4	59.22	66.94	4	61.33	68.95	4	63.52	71.01
3	59.26	66.99	3	61.38	68.99	3	63.57	71.05
2	59.30	67.03	2	61.42	69.03	2	63.61	71.09
1	59.35	67.07	1	61.46	69.07	1	63.65	71.13
0	59.39	67.11	0	61.50	69.11	0	63.70	71.17
0.8969	59.43	67.15	0.8919	61.54	69.15	0.8869	63.74	71.22
8	59.48	67.19	8	61.58	69.19	8	63.78	71.26
7	59.52	67.24	7	61.63	69.22	7	63.83	71.30
6	59.57	67.28	6	61.67	69.26	6	63.87	71.34
5	59.61	67.32	5	61.71	69.30	5	63.91	71.38
4	59.65	67.36	4	61.75	69.34	4	63.96	71.42
3	59.70	67.40	3	61.79	69.38	3	64.00	71.46
2	59.74	67.44	2	61.83	69.42	2	64.04	71.50
1	59.78	67.49	1	61.88	69.46	1	64.09	71.54
0	59.83	67.53	0	61.92	69.50	0	64.13	71.58
0.8959	59.87	67.57	0.8909	61.96	69.54	0.8859	64.17	71.62
8	59.91	67.61	8	62.00	69.58	8	64.22	71.66
7	59.96	67.65	7	62.05	69.62	7	64.26	71.70
6	60.00	67.69	6	62.09	69.66	6	64.30	71.74
5	60.04	67.73	5	62.14	69.71	5	64.35	71.78
4	60.08	67.77	4	62.18	69.75	4	64.39	71.82
3	60.13	67.81	3	62.23	69.79	3	64.43	71.86
2	60.17	67.85	2	62.27	69.84	2	64.48	71.90
1	60.21	67.89	1	62.32	69.88	1	64.52	71.94
0	60.26	67.93	0	62.36	69.92	0	64.57	71.98
0.8949	60.29	67.97	0.8899	62.41	69.96	0.8849	64.61	72.02
8	60.33	68.01	8	62.45	70.01	8	64.65	72.06

SPECIFIC GRAVITY AND PERCENTAGE OF ALCOHOL—(*Continued*).

Spec. Grav. at 15.6° C.	Absolute Alcohol.		Spec. Grav. at 15.6° C.	Absolute Alcohol.		Spec. Grav. at 15.6° C.	Absolute Alcohol.	
	Per Cent by Weight.	Per Cent by Volume.		Per Cent by Weight.	Per Cent by Volume.		Per Cent by Weight.	Per Cent by Volume.
0.8829	65.46	72.80	0.8779	67.58	74.74	0.8729	69.67	76.61
8	65.50	72.84	8	67.63	74.78	8	69.71	76.65
7	65.54	72.88	7	67.67	74.82	7	69.75	76.68
6	65.58	72.92	6	67.71	74.86	6	69.79	76.72
5	65.63	72.96	5	67.75	74.89	5	69.83	76.76
4	65.67	72.99	4	67.79	74.93	4	69.88	76.80
3	65.71	73.03	3	67.83	74.97	3	69.92	76.83
2	65.75	73.07	2	67.88	75.01	2	69.96	76.87
1	65.79	73.11	1	67.92	75.04	1	70.00	76.91
0	65.83	73.15	0	67.96	75.08	0	70.04	76.94
0.8819	65.88	73.19	0.8769	68.00	75.12	0.8719	70.08	76.98
8	65.92	73.22	8	68.04	75.16	8	70.12	77.01
7	65.96	73.26	7	68.08	75.19	7	70.16	77.05
6	66.00	73.30	6	68.13	75.23	6	70.20	77.08
5	66.04	73.34	5	68.17	75.27	5	70.24	77.12
4	66.09	73.38	4	68.21	75.30	4	70.28	77.15
3	66.13	73.42	3	68.25	75.34	3	70.32	77.19
2	66.17	73.46	2	68.29	75.38	2	70.36	77.22
1	66.22	73.50	1	68.33	75.42	1	70.40	77.25
0	66.26	73.54	0	68.38	75.45	0	70.44	77.29
0.8809	66.30	73.57	0.8759	68.42	75.49	0.8709	70.48	77.32
8	66.35	73.61	8	68.46	75.53	8	70.52	77.36
7	66.39	73.65	7	68.50	75.57	7	70.56	77.39
6	66.43	73.69	6	68.54	75.60	6	70.60	77.43
5	66.48	73.73	5	68.58	75.64	5	70.64	77.46
4	66.52	73.77	4	68.63	75.68	4	70.68	77.50
3	66.57	73.81	3	68.67	75.72	3	70.72	77.53
2	66.61	73.85	2	68.71	75.75	2	70.76	77.57
1	66.65	73.89	1	68.75	75.79	1	70.80	77.60
0	66.70	73.93	0	68.79	75.83	0	70.84	77.64
0.8799	66.74	73.97	0.8749	68.83	75.87	0.8699	70.88	77.67
8	66.78	74.01	8	68.88	75.90	8	70.92	77.71
7	66.83	74.05	7	68.92	75.94	7	70.96	77.74
6	66.87	74.09	6	68.96	75.98	6	71.00	77.78
5	66.91	74.13	5	69.00	76.01	5	71.04	77.82
4	66.96	74.17	4	69.04	76.05	4	71.08	77.85
3	67.00	74.22	3	69.08	76.09	3	71.13	77.89
2	67.04	74.25	2	69.13	76.13	2	71.17	77.93
1	67.08	74.29	1	69.17	76.16	1	71.21	77.96
0	67.13	74.33	0	69.21	76.20	0	71.25	78.00
0.8789	67.17	74.37	0.8739	69.25	76.24	0.8689	71.29	78.04
8	67.21	74.40	8	69.29	76.27	8	71.33	78.07
7	67.25	74.44	7	69.33	76.31	7	71.38	78.11
6	67.29	74.48	6	69.38	76.35	6	71.42	78.14
5	67.33	74.52	5	69.42	76.39	5	71.46	78.18
4	67.38	74.55	4	69.46	76.42	4	71.50	78.22
3	67.42	74.59	3	69.50	76.46	3	71.54	78.25
2	67.46	74.63	2	69.54	76.50	2	71.58	78.29
1	67.50	74.67	1	69.58	76.53	1	71.63	78.33
0	67.54	74.70	0	69.63	76.57	0	71.67	78.36

SPECIFIC GRAVITY AND PERCENTAGE OF ALCOHOL—(*Continued*).

Spec. Grav. at 15.6° C.	Absolute Alcohol.		Spec. Grav. at 15.6° C.	Absolute Alcohol.		Spec. Grav. at 15.6° C.	Absolute Alcohol.	
	Per Cent by Weight.	Per Cent by Volume.		Per Cent by Weight.	Per Cent by Volume.		Per Cent by Weight.	Per Cent by Volume.
0.8679	71.71	78.40	0.8629	73.83	80.26	0.8579	76.08	82.23
8	71.75	78.44	8	73.88	80.30	8	76.13	82.26
7	71.79	78.47	7	73.92	80.33	7	76.17	82.30
6	71.83	78.51	6	73.96	80.37	6	76.21	82.33
5	71.88	78.55	5	74.00	80.40	5	76.25	82.37
4	71.92	78.58	4	74.05	80.44	4	76.29	82.40
3	71.96	78.62	3	74.09	80.48	3	76.33	82.44
2	72.00	78.66	2	74.14	80.52	2	76.38	82.47
1	72.04	78.70	1	74.18	80.56	1	76.42	82.51
0	72.09	78.73	0	74.23	80.60	0	76.46	82.54
0.8669	72.13	78.77	0.8619	74.27	80.64	0.8569	76.50	82.58
8	72.17	78.81	8	74.32	80.68	8	76.54	82.61
7	72.22	78.85	7	74.36	80.72	7	76.58	82.65
6	72.26	78.89	6	74.41	80.76	6	76.63	82.69
5	72.30	78.93	5	74.45	80.80	5	76.67	82.72
4	72.35	78.96	4	74.50	80.84	4	76.71	82.76
3	72.39	79.00	3	74.55	80.88	3	76.75	82.79
2	72.43	79.04	2	74.59	80.92	2	76.79	82.83
1	72.48	79.08	1	74.64	80.96	1	76.83	82.86
0	72.52	79.12	0	74.68	81.00	0	76.88	82.90
0.8659	72.57	79.16	0.8609	74.73	81.04	0.8559	76.92	82.93
8	72.61	79.19	8	74.77	81.08	8	76.96	82.97
7	72.65	79.23	7	74.82	81.12	7	77.00	83.00
6	72.70	79.27	6	74.86	81.16	6	77.04	83.04
5	72.74	79.31	5	74.91	81.20	5	77.08	83.07
4	72.78	79.35	4	74.95	81.24	4	77.13	83.11
3	72.83	79.39	3	75.00	81.28	3	77.17	83.14
2	72.87	79.42	2	75.05	81.32	2	77.21	83.18
1	72.91	79.46	1	75.09	81.36	1	77.25	83.21
0	72.96	79.50	0	75.14	81.40	0	77.29	83.25
0.8649	73.00	79.54	0.8599	75.18	81.44	0.8549	77.33	83.28
8	73.04	79.57	8	75.23	81.48	8	77.38	83.32

SPECIFIC GRAVITY AND PERCENTAGE OF ALCOHOL—(*Continued*).

Spec. Grav. at 15.6° C.	Absolute Alcohol. Per Cent by Weight.	Absolute Alcohol. Per Cent. by Volume.	Spec. Grav. at 15.6° C.	Absolute Alcohol. Per Cent by Weight.	Absolute Alcohol. Per Cent by Volume.	Spec. Grav. at 15.6° C.	Absolute Alcohol. Per Cent by Weight.	Absolute Alcohol. Per Cent by Volume.
0.8529	78.16	83.98	0.8479	80.17	85.63	0.8429	82.19	87.27
8	78.20	84.01	8	80.21	85.66	8	82.23	87.30
7	78.24	84.04	7	80.25	85.70	7	82.27	87.34
6	78.28	84.08	6	80.29	85.73	6	82.31	87.37
5	78.32	84.11	5	80.33	85.77	5	82.35	87.40
4	78.36	84.14	4	80.38	85.80	4	82.38	87.43
3	78.40	84.18	3	80.42	85.84	3	82.42	87.46
2	78.44	84.21	2	80.46	85.87	2	82.46	87.49
1	78.48	84.24	1	80.50	85.90	1	82.50	87.52
0	78.52	84.27	0	80.54	85.94	0	82.54	87.55
0.8519	78.56	84.31	0.8469	80.58	85.97	0.8419	82.58	87.58
8	78.60	84.34	8	80.63	86.01	8	82.62	87.61
7	78.64	84.37	7	80.67	86.04	7	82.65	87.64
6	78.68	84.41	6	80.71	86.08	6	82.69	87.67
5	78.72	84.44	5	80.75	86.11	5	82.73	87.70
4	78.76	84.47	4	80.79	86.15	4	82.77	87.73
3	78.80	84.51	3	80.83	86.18	3	82.81	87.76
2	78.84	84.54	2	80.88	86.22	2	82.85	87.79
1	78.88	84.57	1	80.92	86.25	1	82.88	87.82
0	78.92	84.60	0	80.96	86.28	0	82.92	87.85
0.8509	78.96	84.64	0.8459	81.00	86.32	0.8409	82.96	87.88
8	79.00	84.67	8	81.04	86.35	8	83.00	87.91
7	79.04	84.70	7	81.08	86.38	7	83.04	87.94
6	79.08	84.74	6	81.12	86.42	6	83.08	87.97
5	79.12	84.77	5	81.16	86.45	5	83.12	88.00
4	79.16	84.80	4	81.20	86.48	4	83.15	88.03
3	79.20	84.83	3	81.24	86.51	3	83.19	88.06
2	79.24	84.87	2	81.28	86.54	2	83.23	88.09
1	79.28	84.90	1	81.32	86.58	1	83.27	88.13
0	79.32	84.93	0	81.36	86.61	0	83.31	88.16
0.8499	79.36	84.97	0.8449	81.40	86.64	0.8399	83.35	88.19
8	79.40	85.00	8	81.44	86.67	8	83.38	88.22
7	79.44	85.03	7	81.48	86.71	7	83.42	88.25
6	79.48	85.06	6	81.52	86.74	6	83.46	88.28
5	79.52	85.10	5	81.56	86.77	5	83.50	88.31
4	79.56	85.13	4	81.60	86.80	4	83.54	88.34
3	79.60	85.16	3	81.64	86.83	3	83.58	88.37
2	79.64	85.19	2	81.68	86.87	2	83.62	88.40
1	79.68	85.23	1	81.72	86.90	1	83.65	88.43
0	79.72	85.26	0	81.76	86.93	0	83.69	88.46
0.8489	79.76	85.29	0.8439	81.80	86.96	0.8389	83.73	88.49
8	79.80	85.33	8	81.84	86.99	8	83.77	88.52
7	79.84	85.36	7	81.88	87.03	7	83.81	88.55
6	79.88	85.39	6	81.92	87.06	6	83.85	88.58
5	79.92	85.42	5	81.96	87.09	5	83.88	88.61
4	79.96	85.46	4	82.00	87.12	4	83.92	88.64
3	80.00	85.49	3	82.04	87.15	3	83.96	88.67
2	80.04	85.53	2	82.08	87.18	2	84.00	88.70
1	80.08	85.56	1	82.12	87.21	1	84.04	88.73
0	80.13	85.59	0	82.15	87.24	0	84.08	88.76

SPECIFIC GRAVITY AND PERCENTAGE OF ALCOHOL—(*Continued*).

Spec. Grav. at 15.6° C.	Absolute Alcohol.		Spec. Grav. at 15.6° C.	Absolute Alcohol.		Spec. Grav. at 15.6° C.	Absolute Alcohol.	
	Per Cent by Weight.	Per Cent by Volume.		Per Cent by Weight.	Per Cent by Volume.		Per Cent by Weight.	Per Cent. by Volume.
0.8379	84.12	88.79	0.8329	86.08	90.32	0.8279	88.00	91.78
8	84.16	88.83	8	86.12	90.35	8	88.04	91.81
7	84.20	88.86	7	86.15	90.38	7	88.08	91.84
6	84.24	88.89	6	86.19	90.40	6	88.12	91.87
5	84.28	88.92	5	86.23	90.43	5	88.16	91.90
4	84.32	88.95	4	86.27	90.46	4	88.20	91.93
3	84.36	88.98	3	86.31	90.49	3	88.24	91.96
2	84.40	89.01	2	86.35	90.52	2	88.28	91.99
1	84.44	89.05	1	86.38	90.55	1	88.32	92.02
0	84.48	89.08	0	86.42	90.58	0	88.36	92.05
0.8369	84.52	89.11	0.8319	86.46	90.61	0.8269	88.40	92.08
8	84.56	89.14	8	86.50	90.64	8	88.44	92.12
7	84.60	89.17	7	86.54	90.67	7	88.48	92.15
6	84.64	89.20	6	86.58	90.70	6	88.52	92.18
5	84.68	89.24	5	86.62	90.73	5	88.56	92.21
4	84.72	89.27	4	86.65	90.76	4	88.60	92.24
3	84.76	89.30	3	86.69	90.79	3	88.64	92.27
2	84.80	89.33	2	86.73	90.82	2	88.68	92.30
1	84.84	89.36	1	86.77	90.85	1	88.72	92.33
0	84.88	89.39	0	86.81	90.88	0	88.76	92.36
0.8359	84.92	89.42	0.8309	86.85	90.90	0.8259	88.80	92.39
8	84.96	89.46	8	86.88	90.93	8	88.84	92.42
7	85.00	89.49	7	86.92	90.96	7	88.88	92.45
6	85.04	89 52	6	86.96	90.99	6	88.92	92.48
5	85.08	89.55	5	87.00	91.02	5	88.96	92.51
4	85.12	89.58	4	87.04	91.05	4	89.00	92.54
3	85.15	89.61	3	87.08	91.08	3	89.04	92.57
2	85.19	89.64	2	87.12	91.11	2	89.08	92.60
1	85.23	89.67	1	87.15	91.14	1	89.12	92.63
0	85.27	89.70	0	87.19	91.17	0	89.16	92.66
0.8349	85.31	89.72	0.8299	87.23	91.20	0.8249	89.19	92.68
8	85.35	89.75	8	87.27	91.23	8	89.23	92.71
7	85.38	89.78	7	87.31	91.25	7	89.27	92.74

SPECIFIC GRAVITY AND PERCENTAGE OF ALCOHOL—(*Continued*).

Spec. Grav. at 15.6° C.	Absolute Alcohol.		Spec. Grav. at 15.6° C.	Absolute Alcohol.		Spec. Grav. at 15.6° C.	Absolute Alcohol.	
	Per Cent by Weight.	Per Cent by Volume.		Per Cent by Weight.	Per Cent by Volume.		Per Cent by Weight.	Per Cent by Volume.
0.8229	89.96	93.26	0.8179	91.75	94.53	0.8129	93.59	95.84
8	90.00	93.29	8	91.79	94.56	8	93.63	95.87
7	90.04	93.31	7	91.82	94.59	7	93.67	95.90
6	90.07	93.34	6	91.86	94.61	6	93.70	95.92
5	90.11	93.36	5	91.89	94.64	5	93.74	95.95
4	90.14	93.39	4	91.93	94.66	4	93.78	95.97
3	90.18	93.41	3	91.96	94.69	3	93.81	96.00
2	90.21	93.44	2	92.00	94.71	2	93.85	96.03
1	90.25	93.47	1	92.04	94.74	1	93.89	96.05
0	90.29	93.49	0	92.07	94.76	0	93.92	96.08
0.8219	90.32	93.52	0.8169	92.11	94.79	0.8119	93.96	96.11
8	90.36	93.74	8	92.15	94.82	8	94.00	96.13
7	90.39	93.57	7	92.18	94.84	7	94.03	96.16
6	90.43	93.59	6	92.22	94.87	6	94.07	96.18
5	90.46	93.62	5	92.26	94.90	5	94.10	96.20
4	90.50	93.64	4	92.30	94.92	4	94.14	96.22
3	90.54	93.67	3	92.33	94.95	3	94.17	96.25
2	90.57	93.70	2	92.37	94.98	2	94.21	96.27
1	90.61	93.72	1	92.41	95.00	1	94.24	96.29
0	90.64	93.75	0	92.44	95.03	0	94.28	96.32
0.8209	90.68	93.77	0.8159	92.48	95.06	0.8109	94.31	96.34
8	90.71	93.80	8	92.52	95.08	8	94.34	96.36
7	90.75	93.82	7	92.55	95.11	7	94.38	96.39
6	90.79	93.85	6	92.59	95.13	6	94.41	96.41
5	90.82	93.87	5	92.63	95.16	5	94.45	96.43
4	90.86	93.90	4	92.67	95.19	4	94.48	96.46
3	90.89	93.93	3	92.70	95.21	3	94.52	96.48
2	90.93	93.95	2	92.74	95.24	2	94.55	96.50
1	90.96	93.98	1	92.78	95.27	1	94.59	96.53
0	91.00	94.00	0	92.81	95.29	0	94.62	96.55
0.8199	91.04	94.03	0.8149	92.85	95.32	0.8099	94.65	96.57
8	91.07	94.05	8	92.89	95.35	8	94.68	96.60
7	91.11	94.08	7	92.92	95.37	7	94.73	96.62
6	91.14	94.10	6	92.96	95.40	6	94.76	96.64
5	91.18	94.13	5	93.00	95.42	5	94.80	96.67
4	91.21	94.15	4	93.04	95.45	4	94.83	96.69
3	91.25	94.18	3	93.07	95.48	3	94.86	96.71
2	91.29	94.21	2	93.11	95.50	2	94.90	96.74
1	91.32	94.23	1	93.15	95.53	1	94.93	96.76
0	91.36	94.26	0	93.18	95.55	0	94.97	96.78
0.8189	91.39	94.28	0.8139	93.22	95.58	0.8089	95.00	96.80
8	91.43	94.31	8	93.26	95.61	8	95.04	96.83
7	91.46	94.33	7	93.30	95.63	7	95.07	96.85
6	91.50	94.36	6	93.33	95.66	6	95.11	96.88
5	91.54	94.38	5	93.37	95.69	5	95.14	96.90
4	91.57	94.41	4	93.41	95.71	4	95.18	96.93
3	91.61	94.43	3	93.44	95.74	3	95.21	96.95
2	91.64	94.46	2	93.48	95.76	2	95.25	96.98
1	91.68	94.48	1	93.52	95.79	1	95.29	97.00
0	91.71	94.51	0	93.55	95.82	0	95.32	97.02

SPECIFIC GRAVITY AND PERCENTAGE OF ALCOHOL—(*Continued*).

Spec. Grav at 15.6° C.	Absolute Alcohol.		Spec. Grav. at 15.6° C.	Absolute Alcohol.		Spec. Grav. at 15.6° C.	Absolute Alcohol.	
	Per Cent by Weight.	Per Cent by Volume.		Per Cent by Weight.	Per Cent by Volume.		Per Cent by Weight.	Per Cent by Volume.
0.8079	95.36	97.05	0.8029	97.07	98.18	0.7979	98.69	99.18
	95.39	97.07	8	97.10	98.20	8	98.72	99.20
7	95.43	97.10	7	97.13	98.22	7	98.75	99.22
6	95.46	97.12	6	97.16	98.24	6	98.78	99.24
5	95.50	97.15	5	97.20	98.27	5	98.81	99.26
4	95.54	97.17	4	97.23	98.29	4	98.84	99.27
3	95.57	97.20	3	97.26	98.31	3	98.87	99.29
2	95.61	97.22	2	97.30	98.33	2	98.91	99.31
1	95.64	97.24	1	97.33	98.35	1	98.94	99.33
0	95.68	97.27	0	97.37	98.37	0	98.97	99.35
0.8069	95.71	97.29	0.8019	97.40	98.39	0.7969	99.00	99.37
8	95.75	97.32	8	97.43	98.42	8	99.03	99.39
7	95.79	97.34	7	97.46	98.44	7	99.06	99.41
6	95.82	97.37	6	97.50	98.46	6	99.10	99.43
5	95.86	97.39	5	97.53	98.48	5	99.13	99.45
4	95.89	97.41	4	97.57	98.50	4	99.16	99.47
3	95.93	97.44	3	97.60	98.52	3	99.19	99.49
2	95.96	97.46	2	97.63	98.54	2	99.23	99.51
1	96.00	97.49	1	97.66	98.56	1	99.26	99.53
0	96.03	97.51	0	97.70	98.59	0	99.29	99.55
0.8059	96.07	97.53	0.8009	97.73	98.61	0.7959	99.32	99.57
8	96.10	97.55	8	97.76	98.63	8	99.36	99.59
7	96.13	97.57	7	97.80	98.65	7	99.39	99.61
6	96.16	97.60	6	97.83	98.67	6	99.42	99.63
5	96.20	97.62	5	97.87	98.69	5	99.45	99.65
4	96.23	97.64	4	97.90	98.71	4	99.48	99.67
3	96.26	97.66	3	97.93	98.74	3	99.52	99.69
2	96.30	97.68	2	97.96	98.76	2	99.55	99.71
1	96.33	97.70	1	98.00	98.78	1	99.58	99.73
0	96.37	97.73	0	98.03	98.80	0	99.61	99.75
0.8049	96.40	97.75	0.7999	98.06	98.82	0.7949	99.65	99.77
8	96.43	97.77	8	98.09	98.83	8	99.68	99.80
7	96.46	97.79	7	98.13	98.85	7	99.71	99.82



(4) Determination of Alcohol by the Ebullioscope or Vaporimeter is based on the variation in boiling-point of mixtures of alcohol and water, in accordance with the amount of alcohol present. There are various forms of this instrument, one of the simplest and most convenient being that of Salleron, Fig. 112, the apparatus being known in France as an

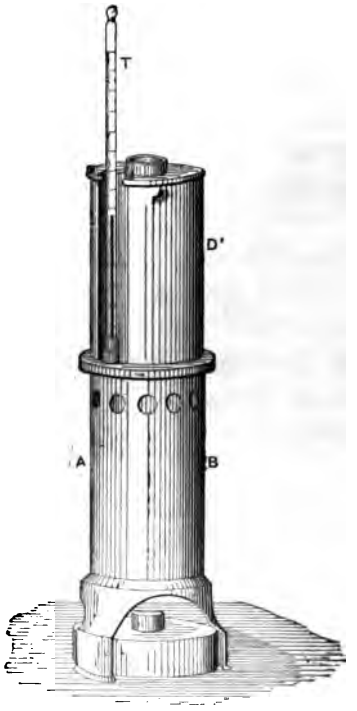


FIG. 112.—Salleron's Ebullioscope.



FIG. 113.—Ebullioscope Scale.

ebulliometer. This consists of a jacketed metallic reservoir, heated by a lamp placed beneath, and fitted with a return-flow condenser at the top and with a delicate thermometer graduated in tenths of a degree.

As the boiling-point of water varies with the atmospheric pressure, it is necessary to determine the actual boiling-point corresponding with the barometric conditions each time a series of determinations are made.

This is done by boiling a measured portion of distilled water in the reservoir, and carefully noting the temperature when it becomes constant.

The reservoir is then rinsed out with a little of the liquor to be tested, after which a measured amount of this liquor is boiled in the reservoir and the temperature again noted. A sliding scale (Fig. 113) accompanies the instrument, having three graduated parts as shown. The central movable portion is graduated in degrees and tenths of a degree centigrade, the part at the left has the per cent of alcohol corresponding to the temperature in the case of simple mixtures of alcohol and water, while the part at the right is used for reading the per cent in the case of wine, cider, beer, etc., which have a considerable residue. The movable scale bearing the degrees of temperature is first set with the actual temperature of boiling water (as ascertained) opposite the 0 mark on the stationary scale. Suppose the temperature of boiling water has been found to be 100.1°. The scale is in this case set as shown in Fig. 113. Suppose also the temperature of boiling of the wine to be tested is found to be 89.3°. From the right-hand scale the corresponding per cent of alcohol is found to be 17.2.

When the liquor to be tested contains more than 25% of alcohol, it is necessary to dilute with a measured amount of distilled water and calculate the per cent from the dilution.

When once the boiling-point of water has been determined for a given barometric pressure, it is unnecessary to change the position of the sliding scale during a series of alcohol determinations unless that pressure changes.

Expression of Results.—Some confusion is caused by the three ways of expressing results of the alcohol determination, whether as per cent by



100 cc. and, in order to have a common basis of comparison between the composition of American and of European wines, this manner of expression has to some extent been adopted in the United States.

Still another mode of expression, and a cumbersome one, is that of the English excise, wherein the alcoholic strength is referred for a standard to so-called "proof spirit," fixed by Parliament as a liquid of specific gravity 0.91984 at 15.5° C., which accordingly contains 49.24% by weight or 57.06% by volume of absolute alcohol. If a liquor is stronger in alcohol than this, it is said to be overproof, and if weaker, underproof. Its strength is further expressed in degrees over or under, water being regarded as 100° underproof. By liquor of 20° underproof, for instance, is meant that it consists of 80 parts by volume of proof-spirit and 20 parts by volume of water at 15.5° C., while by 20° overproof is meant that at that temperature 100 parts by volume of the liquor has to be diluted to 120 parts to yield proof spirit.

To calculate the per cent by volume of proof spirit from the per cent of alcohol by volume, divide the latter by 0.5706 or multiply it by 1.7525.

Direct Determination of Extract.—In liquors having a high sugar content, the extract or total solids cannot be determined accurately by evaporation at the temperature of boiling water, owing to the dehydration of the reducing sugars at temperatures exceeding 75°. When extreme accuracy is required, such liquors should be dried *in vacuo* at 75°, or in a McGill oven (p. 481).

Approximate results satisfactory in most cases are obtained by heating for two and one-half hours 10 grams of the liquor in a tared platinum dish at the temperature of boiling water. If the results are to be expressed in grams per 100 cc., instead of weighing out 10 grams, 10 cc. of the liquor are measured by a pipette into a tared dish. With distilled liquors having low residues, accurate results are obtainable by direct evaporation at 100°, using preferably 25 grams or 25 cc. according as the result is to be expressed in per cent by weight or grams per 100 cc.

Extract in wine and beer is more readily calculated indirectly from their specific gravity as noted elsewhere.

Determination of Ash.—The residue from the determination of the extract is incinerated to a white ash in the original dish at a low red heat, either over a Bunsen flame or in a muffle. The dish is finally cooled in a desiccator and weighed.

Preservatives and Artificial Sweeteners in liquors are identified as described in Chapters XVII and XVIII.

FERMENTED LIQUORS.

The fermented juices of many varieties of fruits and berries furnish beverages more or less popular in various localities, especially for home consumption, though, with the exception of the products of the apple and the grape, few of them are found on the market. The following table shows the average percentage of sugar and free acid in the expressed juice or must of fruits, according to Fresenius, arranged in the order of their sugar content:

	Per Cent Sugar.	Per Cent Free Acid as Malic.
Peaches	1.99	0.85
Apricots	2.13	1.29
Plums	2.80	1.72
Green gages	4.18	0.67
Raspberries	4.84	1.80
Blackberries	5.32	1.42
Strawberries	6.89	1.57
Currants	7.30	2.43
German prunes	7.56	1.08
Gooseberries	8.00	1.63
Pears	8.43	0.09
Apples	9.14	0.82
Mulberries	10.00	2.02
Sour cherries	10.44	1.52
Sweet cherries	15.30	0.88
Grapes	16.15	0.80

CIDER.

Cider is the expressed juice of the apple. When fresh and before fermentation has set in, it is known as sweet cider, but it does not long remain in this condition, developing after a good fermentation from 3 to

washed off. As a rule the apples commonly used by farmers for cider-making are those that are unsalable or unfit for other purposes, being chiefly windfalls or bruised and imperfect fruit. The apples are usually first crushed in a mill to a coarse pulp, which is afterward subjected to pressure in a suitable press and the juice thus extracted.

In this country but little attention is paid to the after processes, the juice being usually transferred directly to barrels, which are not always particularly clean, and allowed to ferment spontaneously in a convenient place, subject to changes in temperature. There is little wonder that cider so made will keep but a short time and quickly goes over into vinegar, unless salicylic acid or other antiseptic is added.

In France more care is taken to regulate the temperature of fermentation, to insure absolute cleanness of all receptacles, and to separate out contaminating impurities. A preliminary fermentation is usually given to the juice in open vats, during which the yeast spores are developed, while impurities separate out both by rising to the surface and by settling to the bottom, care being taken to avoid the development of acetic fermentation. At the proper time the juice is "racked off" or drawn from the clear portion between the top and bottom, transferred to scrupulously clean barrels, and allowed to undergo a second fermentation at a lower temperature than before.

Sometimes the "racking off" is repeated, and the juice is further clarified by "fining" or treating with isinglass, which carries down certain albuminous substances.

Cider thus made is capable of keeping a very long time.

In England cider is sometimes "fined" by treatment with milk, one quart of the latter being added to eighteen gallons of cider.

The apple pomace, left as a residue, is generally steeped in water and repressed. The juice from the second pressing is occasionally added to the first for cider manufacture, but more often is concentrated and made into apple jelly, or used as a fortifier for vinegar to make up deficiency in solids.

Composition of Cider. The following tables, due to Browne,* show the chemical composition of the freshly expressed juice of several American varieties of apple, as well as that of a few fermented samples of cider of known purity.

* Penn. Dept. of Agric., Bul. 58.

APPLE JUICES.

	Specific Gravity.	Solids.	Invert Sugar.	Sucrose.	Total Sugar.	Total Sugar after Inversion.	Free Malic Acid.	Ash.	Undetermined, Pectin, etc.	Rotation, 400-mm. Tube, Ventzke Scale. Degrees to the Left.
Red astrachan.....	1.05317	11.78	6.87	3.63	10.50	10.69	1.14	0.37	0.77	23.72
Early harvest.....	1.05522	13.29	7.49	3.97	11.46	11.67	0.90	0.28	0.65	24.32
Yellow transparent.	1.05020	11.71	8.03	2.10	10.14	10.24	0.86	0.27	0.44	
Early strawberry...	1.04949	11.81	5.47	4.21	9.68	9.00	0.78	0.24	1.11	19.24
Sweet bough.....	1.04979	11.87	7.61	3.08	10.69	10.85	0.10	.....	.....	39.40
Baldwin, green....	1.04882	11.36	6.96	1.63	8.59	8.68	1.24	0.31	1.22	36.16
" ripe.....	1.07362	16.82	7.97	7.05	15.02	15.39	0.67	0.26	0.87	
Ben Davis.....	1.05389	12.77	7.11	3.85	10.96	11.16	0.46	0.28	1.07	49.00
Bellflower.....	1.06270	14.90	9.06	4.32	13.38	13.61	0.58	0.28	0.66	39.20
Tulpahocken.....	1.05727	13.94	9.68	3.11	12.79	12.95	0.26	0.24	0.49	48.20
Unknown.....	1.05901	13.75	10.52	2.31	12.83	12.95	0.44	0.26	0.22	44.18

FERMENTED CIDER (MIXED APPLES).

	Specific Gravity.	Solids.	Invert Sugar.	Malic Acid.	Acetic Acid.	Alcohol.	Pectin.	Ash.	Rotation, 400-mm. Tube, Ventzke Scale. Degrees to the Left.
A...	1.90805	1.94	0.19	0.21	0.24	6.85	0.03	0.25	2.30
B...	1.00122	2.71	0.19	0.24	0.42	5.13	0.03	0.32	2.49
C...	1.00525	3.26	0.89	0.30	0.48	4.67	0.05	0.29	5.28
D..	1.00071	1.93	0.34	0.27	0.21	4.95	0.05	0.23	2.00
E...	1.00512	2.71	0.24	0.29	1.06	4.26	0.06	0.36	1.76



In the municipal laboratory of Paris, Sanglé Ferrière has analyzed eleven samples of known-purity cider with the following results:

	Density.	Per Cent Alcohol by Volume.	Extract per Liter.	Sugar per Liter.		Polarisation, Laurent.	Ash per Liter.	Alkalinity of Ash, as K_2CO_3 per Liter.	Acidity as H_2SO_4.	
				Before Inversion.	After Inversion.				Total.	Fixed.
Mean.....	1.0159	3.9	52.67	21.31	21.62	−4°.26	3.26	2.56	5.27	2.55
Maximum...	1.0410	6.2	114.00	59.40	60.80	−11°.20	4.32	3.68	6.59	2.94
Minimum. ..	1.0012	1.1	22.62	Trace	Trace	0	2.48	2.04	4.20	1.47

Six samples of bottled "sweet" cider purchased in Massachusetts were analyzed in the Food and Drug Laboratory of the Board of Health with the following results:

	Per Cent Alcohol by Weight.	Per Cent Acid as Malic.	Per Cent Extract.
Maximum.....	8.00	0.72	7.82
Minimum.....	3.55	0.48	2.42
Average.....	5.71	0.58	4.19

Browne gives the following as the composition of the mixed ash of several varieties of apple:

Ingredient.	Per-centage.		Ingredient.	Per-centage.
Potash (K_2O).....	55.94	Equivalent to	Potassium carbonate (K_2CO_3)...	6.85
Soda (Na_2O).....	0.31		Potassium phosphate (K_3PO_4)...	14.55
Lime (CaO).....	4.43		Sodium chloride (NaCl).....	0.60
Magnesia (MgO).....	3.78		Calcium sulphate ($CaSO_4$).....	4.52
Oxide of iron (Fe_2O_3).....	0.95		Calcium oxide (CaO).....	2.57
Oxide of aluminum (Al_2O_3).....	0.80		Magnesium phosphate ($Mg_3P_2O_8$)	6.07
Chlorine (Cl).....	0.30		Magnesium oxide (MgO).....	0.59
Silica (SiO_2).....	0.40		Ferric oxide (Fe_2O_3).....	0.95
Sulphuric acid (SO_3).....	2.66		Aluminum oxide (Al_2O_3).....	0.80
Phosphoric acid (P_2O_5).....	8.64		Silica (SiO_2).....	0.40
Carbonic acid (CO_2).....	21.60			
	99.90			
Deduct oxygen equivalent to Cl..	.09			
Total.....	99.81		Total.....	99.80

Burcker * gives the following composition of the ash of cider:

	Per Cent.
Silica	0.94
Phosphoric acid	12.68
Lime	2.77
Magnesia	2.05
Oxides of iron and manganese	0.94
Potash	53.74
Soda	1.10
Carbonic acid	25.78
	100.00

Adulteration of Cider.—The Committee on Standards of the A. O. A. C. have submitted for adoption the following standards for cider: Alcohol not more than 8%, extract not less than 1.8% determined by evaporation in an open vessel at ordinary atmospheric pressure and at the temperature of boiling water; ash not less than 0.2%.

Entirely factitious cider made from other than apple stock is rarely found, though the product as sold is frequently of inferior quality and adulterated. The chief adulterants are water and sugar, and the use of antiseptics is common, especially of salicylic and sulphurous acids, and occasionally beta-naphthol.

Sodium carbonate is sometimes added to cider to neutralize the acid and thus prevent acetic fermentation. An abnormally high ash (say in excess of 0.35%) would point toward the presence of added alkali.

Watering is apparent when the content of alcohol, solids, and ash of the suspected sample are found to be considerably below the corre-



adulterants alleged to be of frequent occurrence in French cider, but not commonly found in this country are commercial glucose, tartaric acid (to increase the acidity of a watered product), and coal-tar colors.

Absence or deficiency of malates is conclusive evidence of fraud, indicating the admixture of notable quantities of the juice of the second pressing of pomace.

Sugar is rendered apparent by the right-handed polarization of the sample, pure cider always polarizing well to the left. If after inversion of a dextro-rotary cider the polarization is still to the right, commercial glucose is indicated; if the reading after inversion is to the left, cane sugar has undoubtedly been added.

Frequently the analyst has only to determine the alcohol, especially in cases of seizure, to ascertain whether or not there has been violation of the liquor laws.

PERRY OR PEAR CIDER.

This is a common French product, but is rarely if ever found on sale in this country, though sometimes made for home consumption. In composition and in method of manufacture it much resembles apple cider. It is also subject to the same forms of adulteration.

The following table summarizes a number of analyses made by Truelle on pear juice, or must, amounts being expressed in parts per thousand:

	Mean.	Maximum.	Minimum.
Specific gravity	1.0845	1.0675	1.0980
Invert sugar	145.64	108.10	200
Sucrose	36.74	16.69	61.41
Total fermentable sugars (as dextrose)	184.14	143.78	220
Tannin	1.78	1.01	3.20
Pectin and albuminous substances	13.08	3	18
Acidity (as sulphuric acid)	1.47	0.76	2.40

The following analysis of champagne perry is taken from the *Lancet* of October 1, 1892:

Alcohol by weight	1.45
Alcohol by volume	1.80
Solids	11.00
Ash	0.35

WINE.

Wine in its broadest sense is the fermented expressed juice of any fruit, though the term, unless otherwise restricted, is generally understood to apply to the juice of the grape.

The organism present in grape juice that plays the chief part in its alcoholic fermentation is the *Saccharomyces ellipsoideus*, a yeast which exists on the skins of the grape.

Process of Manufacture.—The grapes, which should be fully ripe, are picked and sometimes sorted, according to the care that is taken in grading the product. They are also sometimes freed from the stems, which contain considerable tannic acid, and which when crushed with the grapes impart a certain astringency to the final product. The grapes are crushed either by machinery or by the bare feet, and the juice is pressed out from the pulp in various ways, by screw or hydraulic press, or by the centrifugal process.

A certain amount of juice runs off from the preliminary crushing known as the first run, and makes the choicest wine. The product from the pressure constitutes the second run, after which the pomace, by steeping in water and repressing, is made to yield an inferior juice used in vinegar-making.

Red wines are made from dark grapes by fermenting the pulp, before pressing, with the skins, which by this treatment yield up their rich color (*œnocyanin*) to the juice. Besides the color, the skins contain also tannin. White wine is made from the pressed pulp, freed from the skins at once, or from the pulp of white grapes. The unfermented must constitutes from 60 to 80 per cent of the weight of the grape.



liable to occur at this time, are avoided as much as possible by using especially clean casks, which are frequently "sulphured" (or burnt out with sulphur) before being used. The wine is also sometimes clarified, or "fined," by treatment with gelatin, which mechanically removes many impurities by precipitation, or is subjected to pasteurization before finally being bottled or stored in casks.

Classification of Wines.—Wines are either *natural* or *fortified*. Natural wines are those which contain no added sugar or alcohol, but which are exclusively the product of the simple juice, fermented under the best conditions, either till the sugar has been used up, or till the yeast food is exhausted, or until the yeast growth has been checked by the strength of the alcohol developed. When the alcohol content amounts to 14% by weight there can be no further fermentation due to yeast, so that this is the highest limit for natural wine. Examples of natural wines are hock and claret and many California wines.

Fortified wines are those to which alcohol has been added, usually before the natural fermentation has been allowed to proceed to a finish. For this reason considerable sugar is usually left, and such wines are more often sweet. Examples of fortified wines are Madeira, sherry, and port.

Volatile ethers (products of volatile acids) predominate as a rule in natural wines, while fixed ethers (from the fixed acids as tartaric) are most characteristic in fortified wines.

Wines are also variously classified according to characteristic properties possessed by them, as *still* or *sparkling*, *red* or *white*, "*dry*" or *sweet*, etc.

Still wines are those in which there is but little carbon dioxide remaining, so that they do not effervesce. Sparkling wines are more or less heavily charged with carbon dioxide, either naturally, as in the case of champagne, wherein the gas is formed by after-fermentation of added sugar in the corked bottle, or artificially, by carbonating them in a similar manner to "soda-water."

Among the best-known red wines are those of Burgundy and the Bordeaux wines or clarets, while the Rhenish and Moselle wines and the Sauternes are examples of white wine.

"Dry" wines are those in which the sugar has been exhausted by fermentation, while sweet wines possess a considerable amount of unfermented sugar. Whether or not an excess of sugar is left after fermentation has stopped depends upon the amount of yeast food or nitrogenous substance present in the wine. When the proteids are exhausted by the

yeast, fermentation ceases, and for this reason gelatin and other nitrogenous bodies are sometimes added to extend the period of fermentation. Sweet wines are often reinforced by the addition of sugar. Madeira, both red and white, are samples of dry wine, while port wine is one of the sweet variety.

While most of our finer wines still come from France and Germany, large quantities of California wines are now being produced of an extremely high grade and of many varieties.

Composition of Grape Must and of Wine.—König's analyses of a large number of grape musts from different sources are thus summarized:

	Specific Gravity.	Water, Per Cent.	Nitrogenous Material.	Sugar.	Acid.	Other Non-nitrogenous Material.	Ash.
Minimum.	1.0690	51.53	0.11	12.89	0.20	1.68	0.20
Maximum.....	1.2075	82.10	0.57	35.45	1.18	11.62	0.63
Mean.....	1.1024	74.49	0.28	19.71	0.64	4.48	0.40

Typical analyses of German, French, Austrian, Russian, Italian, and Spanish wines are shown in the following table, also due to König:

	Number of Analyses.	Specific Gravity.	Alcohol by Weight.	Extract.	Total Acid as Tartaric.	Free Tartaric Acid.	Cream of Tartar.	Volatile Acid as Acetic.	Sugar.
Germany:									
Moselle.	14	0.9964	7.09	2.24	0.79	.....	.....	.....	0.031
Rhine.	23	1.0005	8.00	2.60	0.81	.....	0.20		
Baden	46		6.65	2.76	0.91	0.218	0.258		

	Glycerin.	Nitrogen.	Ash.	P_2O_5.	Potash.	Lime.	Magnesia.	Sulphuric Acid.	Chlorine.
Germany:									
Moselle	0.72	.....	0.175	0.036	0.068	0.011	0.02	0.012	
Rhine	0.85	.....	0.23	0.046	0.085	0.017	0.022	0.020	
Baden	0.49	.....	0.207	0.025					
Wurtemburg, white wine	0.57	.....	0.25	0.043	0.115	.....	.....	0.009	0.021
" red wine	0.46	.....	0.25	0.040	0.108	0.024	0.010	0.08	
Alsace	0.55	0.028	0.229	0.038					
Lorraine, red wine	0.50	0.019	0.185	0.030	.....	.....	.....	0.008	
France:									
Red wine	0.73	0.043	0.248	0.030	0.106	0.101	0.018	0.033	
White wine	0.97	.....	0.25	0.032	0.098	0.010	0.015	0.038	
Austria:									
Tyrol, red wine	0.65	0.021	0.222	0.027	.....	.....	.....	0.023	
" white wine	0.65	0.020	0.175	0.022	0.077	.....	.....	0.023	
Russia:									
Red wine	0.64	0.036	0.267	0.027	0.111				
White wine	0.59	0.026	0.204	0.030	0.086				
Italy	0.45	.....	0.29	0.032	0.115	0.009	0.017	0.019	
Spain:									
Ordinary red wine	1.09	.....	0.61	0.027	0.242	0.008	.....	0.221	
Sweet wine	0.63	.....	0.74	0.039	0.296	.....	.....	0.212	

On page 558 are given summaries of analyses of American wines compiled from tables of analyses made by Bigelow.*

Varieties of Wine.—*Champagne* is a selected, sweet, white wine, clarified with gelatin, bottled with the addition of cane sugar, mixed with a little brandy, and tightly corked. Sometimes a small amount of yeast is also introduced. Fermentation is allowed to go on at a temperature of about 24° C., during which the wine is highly charged with carbon dioxide. The bottles are set on their side for some months, after which they are inverted till the sediment gathers above the cork, which by careful manipulation is quickly removed so as to throw out the sediment, and is afterward replaced and secured. Champagne contains from 8 to 10 per cent of alcohol and is high in sugar.

Claret is a light, red wine of a deep color, and is somewhat acid and astringent. In alcohol it varies from 8 to 13 per cent by volume. It has very little sugar and is high in volatile ethers.

Madeira is a strong, white wine, possessing a refined, nutty, aromatic flavor when fully aged. It is generally fortified, containing from 17 to 20 per cent of alcohol. It is named from the island which produces it.

Sherry is a deep, amber-colored, sweet, Spanish wine, high in alcohol

* U. S. Dept. of Agric., Bur. of Chem., Bul. 59.

CALIFORNIA WINES.

Specific Gravity.	Per Cent Alcohol by Volume.	Grams per 100 cc.		Glycerin-alcohol Ratio.	Grams per 100 cc.		Polarization.	Grams per 100 cc.				
		Alcohol.	Glycerin.		Total Acids.	Extract.		Reducing Sugar.	Potassium Sulphate.	Proteids.	Tannin and Coloring Matter.	Ash.
.0020	15.09	11.97	.852	8.7:100	.888	3.81	−2.1	.628	.1570	.5544	.358	.429
.9900	8.92	7.09	.330	3.4:100	.368	2.09	−0.5	.040	.0470	.1865	.064	.209
.9970	13.82	10.96	.....	.....	.718	3.34	.....	.....	.....	.....	.349	
.9940	11.00	8.73	.....	.....	.358	2.69	.....	.....	.....	.....	.211	
.9962	15.48	12.29	.656	7:100	.762	3.46	−1.7	.418	.2515	.4482	.328	.416
.9911	8.00	6.35	.461	4.7:100	.408	2.10	−0.5	.030	.0455	.1864	.033	.188
.0050	19.28*	15.30	.....	.....	.834	6.88*	.....	.....	.....	.....	.344	.430
.9900	8.07	6.40	.....	.....	.201	1.91	.....	.....	.....	.....	.050	.202
.0020	14.57	11.57	.971	10.2:100	.788	4.38	−3.5	.626	.1771	.5164	.....	.447
.9883	5.00	3.98	.474	4.6:100	.327	1.51	−0.2	.060	.0633	.0989	.....	.140
.0157	15.20	12.07	.904	7.7:100	.766	6.78	+0.6	3.569	.1648	.3809	.087	.368
.9892	8.23	6.53	.178	1.8:100	.377	1.69	−18.6	.069	.0453	.0867	.015	.050
.9988	22.18	17.60	.918	14:100	.656	4.56	−3.4	.936	.....	.3162	.045	.290
.9882	8.07	6.40	.318	3.4:100	.219	1.09	−0.1	.069	.....	.0988	.034	.148
.0429	22.19	17.61	.797	4.5:100	.700	17.22	−27.1	13.559	.1861	.9379	1.066	.394
.9875	10.38	8.24	.163	1.1:100	.181	2.43	−14.4	.228	.0594	.1893	.059	.222
.0560	21.85	17.34	.936	6.8:100	.798	19.66	−23.1	17.210	.1196	.4751	.350	.436
.9866	8.22	6.52	.324	2:100	.253	1.31	−0.5	.119	.0504	.0859	.021	.156

* Fortified.

(sometimes containing over 20%), being usually fortified. It is slightly acid and possesses much fragrance. Sherry is nearly always "plastered."

Hocks are white German wines, mildly acid, containing 9 to 12 per cent of alcohol by volume. They have very little sugar, and rank among the highest of natural wines. The best-known varieties are Hockheimer and Johanisberger.

Port (*Vinum portense* of the 1870 Pharmacopœia) is a dark-purple, astringent wine, almost always fortified, and hence high in alcohol (from 15 to 18 per cent by volume). It is much improved by aging, during which it looses considerable of its astringency. It contains a large amount of extract, from 2 to 6 per cent of the wine being sugar. The fixed ethers predominate over the volatile.

Standards of Purity for Wine.—The ratio of volatile to fixed acids in pure wine should not exceed 1:3. A higher proportion of volatile acid shows the fact that acetic fermentation has set in.

The presence of any considerable free tartaric acid would indicate the addition of this substance to the wine.

The United States Pharmacopœia has prescribed the following requirements in the case of wines: For white wine (*Vinum album*) the specific gravity at 15.6° should not be less than 0.990 nor more than 1.010; the extract or residue at 100° should not be less than 1.5 nor more than 3%; as indicating the amount of free acid, not less than 3 nor more than 5.2 cc. normal potassium hydroxide should be required to neutralize 50 cc. of the wine, using phenolphthalein as an indicator; it should contain not less than 10 nor more than 14 per cent by weight of absolute alcohol; it should contain only traces of tannin.

For red wine (*Vinum rubum*) the specific gravity at 15.6° should not be less than 0.989 nor more than 1.010; the extract should not be less than 1.6% nor more than 3.5%; its limits as to acidity are the same as with white wine, eosin or fluorescin, however, being used as an indicator; in alcoholic strength, it should, like white wine, come within the limits of 10 and 14 per cent alcohol by weight. It should not be artificially colored, but should show the presence of tannic acid.

The following standards have been recently suggested for adoption by the A. O. A. C. by its Committee on Standards: In white wine the extract should be not less than 1.5 and not more than 4 per cent; in red wine the limits are 1.6 and 4 per cent. For both red and white wine the alcohol should not be more than 16% by volume and the ash not less than 0.14%. For fortified sweet wine the extract should be not less

than 6%, except in case of sherries, which should contain not less than 3%, and the alcohol should not be more than 24 nor less than 14 per cent by volume.

Adulteration of Wine.—Beverages purporting to be wine are sometimes found on sale that are entirely spurious, in that they contain little if any fermented grape juice. Apple cider is not infrequently the basis of such artificial products, and the following recipes given by Brannt may be taken as typical of the composition of these wine substitutes:

Burgundy.—Bring into a barrel 40 quarts of apple juice, 5 pounds of bruised raisins, ½ pound of tartar, 1 quart of bilberry juice, and 3 pounds sugar. Allow the whole to ferment, filling constantly up with cider. Then clarify with isinglass, add about 1 ounce of essence of bitter almonds, and after a few weeks draw off into bottles.

Malaga Wine.—Apple juice, 40 quarts; crushed raisins, 10 pounds; rectified alcohol, 2 quarts; sugar solution, 2 quarts; elderberry flowers, 1 quart; acetic ether, 1 ounce and 2 drachms. The desired coloration is effected by the addition of bilberry or elderberry juice; otherwise the process is the same as given for Burgundy.

Sherry Wine.—Apple juice, 50 quarts; orange-flower water, about 2 drachms; tartar, 2 ounces and 4 drachms; rectified alcohol, 3 quarts; crushed raisins, 10 pounds; acetic ether, 1 ounce and 2 drachms. The process is the same as for Burgundy.

Claret Wine.—Apple juice, 50 quarts; rectified alcohol, 4 quarts; black currant juice, 2 quarts; tartar, 2 ounces and 4 drachms. Color with bilberry juice. The further process is the same as for Burgundy.

Artificial products similar in nature to the above are also mixed in varying proportions with pure wine.

Presence of malates, as well as absence or diminution of total tartaric



the potassium bitartrate present in the wine is, according to Chancel, as follows:

$$2KHC_4H_4O_6 + CaSO_4 = CaC_4H_4O_6 + H_2C_4H_4O_6 + K_2SO_4.$$

Potassium bitartrate	Calcium sulphate	Calcium tartrate	Tartaric acid	Potassium sulphate

Various advantages are said to result from this practice. The wine is clarified by the precipitation of the calcium tartrate, which mechanically carries down with it many impurities, the color of the wine is improved, since the solubility of the coloring principle present in the skins is increased, the fermentation is rendered more rapid and complete, and the keeping qualities of the wine are enhanced. The practice is, however, considered objectionable on account of the potassium sulphate which is left in solution in the wine, and in some countries plastering is forbidden, or the amount of potassium sulphate limited by statute.

The following are analyses of two Spanish wines made from the same grape juice, one of which was plastered. The results are expressed in grams per liter.

	Not Plastered.	Plastered.
Color.	Yellow	Bright red
Extract dried at 100°.	23.3	27.3
Insoluble ash	0.66	0.61
Soluble ash.	2.06	5.38
The soluble ash containing		
Potassium carbonate...	1.29	0.17
" sulphate. ...	0.41	5

The effect of plastering is thus seen to distinctly increase the extract and the soluble ash. Any considerable amount of potassium sulphate is an indication of plastering.

Addition of Cane Sugar.—The term "chaptalizing" is applied in France to the addition to the must of cane sugar for the purpose of increasing the yield in alcohol. The addition of 1,700 grams of sugar to 1,000 liters of must is said to increase the alcoholic strength by 1%. It was formerly customary to add with the sugar calcium carbonate, to partially neutralize the acidity, but this is rarely practiced at present.

The European wine-raising countries are not disposed to regard the reinforcement of wine by added cane sugar in the must as in itself a fraud, unless water is also added, or unless some other form of adulteration is practiced at the same time. In France, however, the addition of cane

sugar is permitted only in wine for local consumption, and is restricted in amount.

The use of commercial glucose in wine instead of cane sugar is not regarded with as much favor, in view of the fact that glucose contains more or less unfermentable matter, and introduces dextrin and various mineral salts into the wine.

To ascertain the nature and extent of the sugars present in wine is frequently of great importance. Much information may be gained from the direct and invert polarization of the sample, as well as from the determination of reducing sugars.

Invert sugar is the only legitimate sugar that should be present in genuine wine. In normal fermentation the dextrose is more quickly destroyed than the levulose, hence the polarization of pure wine is always left-handed, unless all the sugar has been fermented, in which case the reading should be zero.

Seventy-five samples of California red wines, chiefly claret, Burgundy, Rhine, and southern France types, analyzed in the Bureau of Chemistry * of the U. S. Department of Agriculture, polarized from -0.5 to -2.1. Upwards of eighty samples of California white wine (of the types of Burgundy, Sauterne, and southern France) were submitted to polarization and all but four were left-handed. These four (evidently abnormal) polarized from 0. to $+1$. Most of them varied from -0.1 to -3.5.

Thirteen of the port wine types (California) had a left-handed polarization of from -14.7 to -27.1. These apparently contained large quantities of unfermented, inverted cane sugar.

A sharp, right-handed polarization would indicate the presence of either commercial glucose or cane sugar. After inversion, if the reading is still right-handed, glucose is apparent, while if inversion changes the

that the sum of the weight in grams of alcohol in 100 cc. and the total acidity, expressed in grams of sulphuric acid per liter, varies within very narrow limits in pure wines, rarely being below 13 or above 17. A large number of analyses made by Gautier and others would seem to confirm this, so that in the majority of cases, added water would be strongly indicated if the sum of these two constants was materially reduced below 13. It is more conservative to adopt 12.5 as a minimum limit for the sum of the alcohol and total acid expressed as above.

Detection of Added Alcohol.—As a result of the findings of a committee appointed in France to determine the matter of added alcohol, it was submitted that a relation existed between the weight of the extract and that of the alcohol in pure wine. In the case of red wines, if the weight of the alcohol, divided by the weight of the extract (both expressed in grams per 100 cc.) exceeds 4.6, the addition of alcohol is strongly indicated. With white wines, the quotient obtained by dividing the weight of alcohol by weight of extract should not exceed 6.6. If it does, added alcohol is to be suspected.

In the case of plastered wines containing sulphate of potassium, or wines having added sugar, it is necessary to deduct from the total extract the weight of the reducing sugar and of the potassium sulphate as found (less 0.1 gram for each of these substances), the difference, or reduced extract as it is called, being used in this case in obtaining the ratio.

Fruit Wines other than Grape.—Wines mostly of domestic manufacture are sometimes made from small fruits, such as raspberries, strawberries, blackberries, gooseberries, elderberries, and currants, as well as from cherries, plums, and apricots. Wines made from most of these fruits readily undergo acetic fermentation unless antiseptics are added, or unless extreme care is taken in their manufacture and keeping. Frequently mixtures of various fruit juices are made to yield excellent wine. Most of the sour fruits require a liberal admixture of sugar to produce an acceptable wine.

The following analysis of currant wine is due to Fresenius:

Alcohol	10.01%
Free acid	0.79%
Sugar	11.94%
Water	77.26%

The alcoholic content of other fruit wines is thus shown by Brannt:

Gooseberry wine	11.84%	alcohol
Elderberry wine	8.79%	"
Orange wine	11.26%	"

METHODS OF ANALYSIS OF WINE AND CIDER.—For determination of specific gravity, alcohol, extract (by direct method), and ash see pp. 527, 528, and 547.

Calculation of the Extract in Wine.—Attention has already been called to the difficulty in accurately determining the extract of sweet wines gravimetrically by evaporation. An approximate determination of the extract may be obtained by calculation from the specific gravity of the dealcoholized liquor, or one may use for this purpose the tables compiled by Windisch, and based on experiments made on drying wine *in vacuo* at 75° C. In wines high in sugar, containing more than 6% of extract, this method is far more accurate than that of drying at 100°, and is to be recommended.

Evaporate a measured portion of the wine on the water-bath to one-fourth its volume, and dilute with water to exactly the volume measured. Determine the specific gravity of this dealcoholized liquid at 15.6°, and from the following table ascertain the extract corresponding.

Determination of Total Acidity.—Carbonated beverages are first freed from carbon dioxide by agitation as described on page 528, after which 25 cc. of the sample are heated just to the boiling-point and titrated with tenth-normal sodium hydroxide, using in the case of white wine or cider phenolphthalein as an indicator. With red wine delicate litmus-paper should be used. Total acidity is usually expressed, in the case of cider as malic, and of wine as tartaric acid. Each cubic centimeter of tenth-normal alkali corresponds to 0.0067 gram malic, or 0.0075 gram tartaric acid. Some chemists express total acidity in terms of sulphuric acid, each cubic centimeter of tenth-normal alkali being equivalent to 0.0049 gram of sulphuric acid.

EXTRACT IN WINE.

[ACCORDING TO WINDISCH.]

Specific Gravity.	Extract.	Specific Gravity.	Extract.	Specific Gravity.	Extract.	Specific Gravity.	Extract.	Specific Gravity.	Extract.	Specific Gravity.	Extract.
1.0000	0.00	1.0065	1.68	1.0130	3.36	1.0195	5.04	1.0260	6.72	1.0325	8.40
1.0001	0.03	1.0066	1.70	1.0131	3.38	1.0196	5.06	1.0261	6.75	1.0326	8.43
1.0002	0.05	1.0067	1.73	1.0132	3.41	1.0197	5.09	1.0262	6.77	1.0327	8.46
1.0003	0.08	1.0068	1.76	1.0133	3.43	1.0198	5.11	1.0263	6.80	1.0328	8.48
1.0004	0.10	1.0069	1.78	1.0134	3.46	1.0199	5.14	1.0264	6.82	1.0329	8.51
1.0005	0.13	1.0070	1.81	1.0135	3.49	1.0200	5.17	1.0265	6.85	1.0330	8.53
1.0006	0.15	1.0071	1.83	1.0136	3.51	1.0201	5.19	1.0266	6.88	1.0331	8.56
1.0007	0.18	1.0072	1.86	1.0137	3.54	1.0202	5.22	1.0267	6.90	1.0332	8.59
1.0008	0.20	1.0073	1.88	1.0138	3.56	1.0203	5.25	1.0268	6.93	1.0333	8.61
1.0009	0.23	1.0074	1.91	1.0139	3.59	1.0204	5.27	1.0269	6.95	1.0334	8.64
1.0010	0.26	1.0075	1.94	1.0140	3.62	1.0205	5.30	1.0270	6.98	1.0335	8.66
1.0011	0.28	1.0076	1.96	1.0141	3.64	1.0206	5.32	1.0271	7.01	1.0336	8.69
1.0012	0.31	1.0077	1.99	1.0142	3.67	1.0207	5.35	1.0272	7.03	1.0337	8.72
1.0013	0.34	1.0078	2.01	1.0143	3.69	1.0208	5.38	1.0273	7.06	1.0338	8.74
1.0014	0.36	1.0079	2.04	1.0144	3.72	1.0209	5.40	1.0274	7.08	1.0339	8.77
1.0015	0.39	1.0080	2.07	1.0145	3.75	1.0210	5.43	1.0275	7.11	1.0340	8.79
1.0016	0.41	1.0081	2.09	1.0146	3.77	1.0211	5.45	1.0276	7.13	1.0341	8.82
1.0017	0.44	1.0082	2.12	1.0147	3.80	1.0212	5.48	1.0277	7.16	1.0342	8.85
1.0018	0.46	1.0083	2.14	1.0148	3.82	1.0213	5.51	1.0278	7.19	1.0343	8.87
1.0019	0.49	1.0084	2.17	1.0149	3.85	1.0214	5.53	1.0279	7.21	1.0344	8.90
1.0020	0.52	1.0085	2.19	1.0150	3.87	1.0215	5.56	1.0280	7.24	1.0345	8.92
1.0021	0.54	1.0086	2.22	1.0151	3.90	1.0216	5.58	1.0281	7.26	1.0346	8.95
1.0022	0.57	1.0087	2.25	1.0152	3.93	1.0217	5.61	1.0282	7.29	1.0347	8.97
1.0023	0.59	1.0088	2.27	1.0153	3.95	1.0218	5.64	1.0283	7.32	1.0348	9.00
1.0024	0.62	1.0089	2.30	1.0154	3.98	1.0219	5.66	1.0284	7.34	1.0349	9.03
1.0025	0.64	1.0090	2.32	1.0155	4.00	1.0220	5.69	1.0285	7.37	1.0350	9.05
1.0026	0.67	1.0091	2.35	1.0156	4.03	1.0220	5.71	1.0286	7.39	1.0351	9.08
1.0027	0.69	1.0092	2.38	1.0157	4.06	1.0222	5.74	1.0287	7.42	1.0352	9.10
1.0028	0.72	1.0093	2.40	1.0158	4.08	1.0223	5.77	1.0288	7.45	1.0353	9.13
1.0029	0.75	1.0094	2.43	1.0159	4.11	1.0224	5.79	1.0289	7.47	1.0354	9.16
1.0030	0.77	1.0095	2.45	1.0160	4.13	1.0225	5.82	1.0290	7.50	1.0355	9.18
1.0031	0.80	1.0096	2.48	1.0161	4.16	1.0226	5.84	1.0291	7.52	1.0356	9.21
1.0032	0.82	1.0097	2.50	1.0162	4.19	1.0227	5.87	1.0292	7.55	1.0357	9.23
1.0033	0.85	1.0098	2.53	1.0163	4.21	1.0228	5.89	1.0293	7.58	1.0358	9.26
1.0034	0.87	1.0099	2.56	1.0164	4.24	1.0229	5.92	1.0294	7.60	1.0359	9.29
1.0035	0.90	1.0100	2.58	1.0165	4.26	1.0230	5.94	1.0295	7.63	1.0360	9.31
1.0036	0.93	1.0101	2.61	1.0166	4.29	1.0231	5.97	1.0296	7.65	1.0361	9.34
1.0037	0.95	1.0102	2.63	1.0167	4.31	1.0232	6.00	1.0297	7.68	1.0362	9.36
1.0038	0.98	1.0103	2.66	1.0168	4.34	1.0233	6.02	1.0298	7.70	1.0363	9.39
1.0039	1.00	1.0104	2.69	1.0169	4.37	1.0234	6.05	1.0299	7.73	1.0364	9.42
1.0040	1.03	1.0105	2.71	1.0170	4.39	1.0235	6.07	1.0300	7.76	1.0365	9.44
1.0041	1.05	1.0106	2.74	1.0171	4.42	1.0236	6.10	1.0301	7.78	1.0366	9.47
1.0042	1.08	1.0107	2.76	1.0172	4.44	1.0237	6.12	1.0302	7.81	1.0367	9.49
1.0043	1.11	1.0108	2.79	1.0173	4.47	1.0238	6.15	1.0303	7.83	1.0368	9.52
1.0044	1.13	1.0109	2.82	1.0174	4.50	1.0239	6.18	1.0304	7.86	1.0369	9.55
1.0045	1.16	1.0110	2.84	1.0175	4.52	1.0240	6.20	1.0305	7.89	1.0370	9.57
1.0046	1.18	1.0111	2.87	1.0176	4.55	1.0241	6.23	1.0306	7.91	1.0371	9.60
1.0047	1.21	1.0112	2.89	1.0177	4.57	1.0242	6.25	1.0307	7.94	1.0372	9.62
1.0048	1.24	1.0113	2.92	1.0178	4.60	1.0243	6.28	1.0308	7.97	1.0373	9.65
1.0049	1.26	1.0114	2.94	1.0179	4.63	1.0244	6.31	1.0309	7.99	1.0374	9.68
1.0050	1.29	1.0115	2.97	1.0180	4.65	1.0245	6.33	1.0310	8.02	1.0375	9.70
1.0051	1.32	1.0116	3.00	1.0181	4.68	1.0246	6.36	1.0311	8.04	1.0376	9.73
1.0052	1.34	1.0117	3.02	1.0182	4.70	1.0247	6.38	1.0312	8.07	1.0377	9.75
1.0053	1.37	1.0118	3.05	1.0183	4.73	1.0248	6.41	1.0313	8.09	1.0378	9.78
1.0054	1.39	1.0119	3.07	1.0184	4.75	1.0249	6.44	1.0314	8.12	1.0379	9.80
1.0055	1.42	1.0120	3.10	1.0185	4.78	1.0250	6.46	1.0315	8.14	1.0380	9.83
1.0056	1.45	1.0121	3.12	1.0186	4.81	1.0251	6.49	1.0316	8.17	1.0381	9.86
1.0057	1.47	1.0122	3.15	1.0187	4.83	1.0252	6.51	1.0317	8.20	1.0382	9.88
1.0058	1.50	1.0123	3.18	1.0188	4.86	1.0253	6.54	1.0318	8.22	1.0383	9.91
1.0059	1.52	1.0124	3.20	1.0189	4.88	1.0254	6.56	1.0319	8.25	1.0384	9.93
1.0060	1.55	1.0125	3.23	1.0190	4.91	1.0255	6.59	1.0320	8.27	1.0385	9.96
1.0061	1.57	1.0126	3.26	1.0191	4.94	1.0256	6.62	1.0321	8.30	1.0386	9.99
1.0062	1.60	1.0127	3.28	1.0192	4.96	1.0257	6.64	1.0322	8.33	1.0387	10.01
1.0063	1.63	1.0128	3.31	1.0193	4.99	1.0258	6.67	1.0323	8.35	1.0388	10.04
1.0064	1.65	1.0129	3.33	1.0194	5.01	1.0259	6.70	1.0324	8.38	1.0389	10.06

EXTRACT IN WINE—(*Continued*).

Specific Gravity.	Extract.	Specific Gravity.	Extract.	Specific Gravity.	Extract.	Specific Gravity.	Extract.	Specific Gravity.	Extract.	Specific Gravity.	Extract.
1.0300	10.09	1.0455	11.78	1.0520	13.47	1.0585	15.16	1.0650	16.86	1.0715	18.56
1.0301	10.11	1.0456	11.81	1.0521	13.49	1.0586	15.19	1.0651	16.88	1.0716	18.58
1.0302	10.14	1.0457	11.83	1.0522	13.52	1.0587	15.22	1.0652	16.91	1.0717	18.61
1.0303	10.17	1.0458	11.86	1.0523	13.55	1.0588	15.24	1.0653	16.94	1.0718	18.63
1.0304	10.19	1.0459	11.88	1.0524	13.57	1.0589	15.27	1.0654	16.96	1.0719	18.66
1.0305	10.22	1.0460	11.91	1.0525	13.60	1.0590	15.29	1.0655	16.99	1.0720	18.69
1.0306	10.25	1.0461	11.94	1.0526	13.62	1.0591	15.32	1.0656	17.01	1.0721	18.71
1.0307	10.27	1.0462	11.96	1.0527	13.65	1.0592	15.35	1.0657	17.04	1.0722	18.74
1.0308	10.30	1.0463	11.99	1.0528	13.68	1.0593	15.37	1.0658	17.07	1.0723	18.76
1.0309	10.32	1.0464	12.01	1.0529	13.70	1.0594	15.40	1.0659	17.09	1.0724	18.79
1.0400	10.35	1.0465	12.04	1.0530	13.73	1.0595	15.42	1.0660	17.12	1.0725	18.82
1.0401	10.37	1.0466	12.06	1.0531	13.75	1.0596	15.45	1.0661	17.14	1.0726	18.84
1.0402	10.40	1.0467	12.09	1.0532	13.78	1.0597	15.48	1.0662	17.17	1.0727	18.87
1.0403	10.43	1.0468	12.12	1.0533	13.81	1.0598	15.50	1.0663	17.20	1.0728	18.90
1.0404	10.45	1.0469	12.14	1.0534	13.83	1.0599	15.53	1.0664	17.22	1.0729	18.92
1.0405	10.48	1.0470	12.17	1.0535	13.86	1.0600	15.55	1.0665	17.25	1.0730	18.95
1.0406	10.51	1.0471	12.19	1.0536	13.89	1.0601	15.58	1.0666	17.27	1.0731	18.97
1.0407	10.53	1.0472	12.22	1.0537	13.91	1.0602	15.61	1.0667	17.30	1.0732	19.00
1.0408	10.56	1.0473	12.25	1.0538	13.94	1.0603	15.63	1.0668	17.33	1.0733	19.03
1.0409	10.58	1.0474	12.27	1.0539	13.96	1.0604	15.66	1.0669	17.35	1.0734	19.05
1.0410	10.61	1.0475	12.30	1.0540	13.99	1.0605	15.68	1.0670	17.38	1.0735	19.08
1.0411	10.63	1.0476	12.32	1.0541	14.01	1.0606	15.71	1.0671	17.41	1.0736	19.10
1.0412	10.66	1.0477	12.35	1.0542	14.04	1.0607	15.74	1.0672	17.43	1.0737	19.13
1.0413	10.69	1.0478	12.38	1.0543	14.07	1.0608	15.76	1.0673	17.46	1.0738	19.16
1.0414	10.71	1.0479	12.40	1.0544	14.09	1.0609	15.79	1.0674	17.48	1.0739	19.18
1.0415	10.74	1.0480	12.43	1.0545	14.12	1.0610	15.81	1.0675	17.51	1.0740	19.21
1.0416	10.76	1.0481	12.45	1.0546	14.14	1.0611	15.84	1.0676	17.54	1.0741	19.23
1.0417	10.79	1.0482	12.48	1.0547	14.17	1.0612	15.87	1.0677	17.56	1.0742	19.26
1.0418	10.82	1.0483	12.51	1.0548	14.20	1.0613	15.89	1.0678	17.59	1.0743	19.29
1.0419	10.84	1.0484	12.53	1.0549	14.22	1.0614	15.92	1.0679	17.62	1.0744	19.31
1.0420	10.87	1.0485	12.56	1.0550	14.25	1.0615	15.94	1.0680	17.64	1.0745	19.34
1.0421	10.90	1.0486	12.58	1.0551	14.28	1.0616	15.97	1.0681	17.67	1.0746	19.37
1.0422	10.92	1.0487	12.61	1.0552	14.30	1.0617	16.00	1.0682	17.69	1.0747	19.39
1.0423	10.95	1.0488	12.64	1.0553	14.33	1.0618	16.02	1.0683	17.72	1.0748	19.42
1.0424	10.97	1.0489	12.66	1.0554	14.35	1.0619	16.05	1.0684	17.75	1.0749	19.44
1.0425	11.00	1.0490	12.69	1.0555	14.38	1.0620	16.07	1.0685	17.77	1.0750	19.47
1.0426	11.03	1.0491	12.71	1.0556	14.41	1.0621	16.10	1.0686	17.80	1.0751	19.50
1.0427	11.05	1.0492	12.74	1.0557	14.43	1.0622	16.13	1.0687	17.83	1.0752	19.52
1.0428	11.08	1.0493	12.77	1.0558	14.46	1.0623	16.15	1.0688	17.85	1.0753	19.55
1.0429	11.10	1.0494	12.79	1.0559	14.48	1.0624	16.18	1.0689	17.88	1.0754	19.58
1.0430	11.13	1.0495	12.82	1.0560	14.51	1.0625	16.21	1.0690	17.90	1.0755	19.60
1.0431	11.15	1.0496	12.84	1.0561	14.54	1.0626	16.23	1.0691	17.93	1.0756	19.63
1.0432	11.18	1.0497	12.87	1.0562	14.56	1.0627	16.26	1.0692	17.95	1.0757	19.65
1.0433	11.21	1.0498	12.90	1.0563	14.59	1.0628	16.28	1.0693	17.98	1.0758	19.68
1.0434	11.23	1.0499	12.92	1.0564	14.61	1.0629	16.31	1.0694	18.01	1.0759	19.71



EXTRACT IN WINE—(*Continued*).

Specific Gravity.	Extract.	Specific Gravity.	Extract.	Specific Gravity.	Extract.	Specific Gravity.	Extract.	Specific Gravity.	Extract.	Specific Gravity.	Extract.
1.0780	20.26	1.0845	21.96	1.0910	23.67	1.0975	25.38	1.1040	27.09	1.1105	28.81
1.0781	20.28	1.0846	21.99	1.0911	23.70	1.0976	25.41	1.1041	27.12	1.1106	28.83
1.0782	20.31	1.0847	22.02	1.0912	23.72	1.0977	25.43	1.1042	27.15	1.1107	28.86
1.0783	20.34	1.0848	22.04	1.0913	23.75	1.0978	25.46	1.1043	27.17	1.1108	28.88
1.0784	20.36	1.0849	22.07	1.0914	23.77	1.0979	25.49	1.1044	27.20	1.1109	28.91
1.0785	20.39	1.0850	22.09	1.0915	23.80	1.0980	25.51	1.1045	27.22	1.1110	28.94
1.0786	20.41	1.0851	22.12	1.0916	23.83	1.0981	25.54	1.1046	27.25	1.1111	28.96
1.0787	20.44	1.0852	22.15	1.0917	23.85	1.0982	25.56	1.1047	27.27	1.1112	28.99
1.0788	20.47	1.0853	22.17	1.0918	23.88	1.0983	25.59	1.1048	27.30	1.1113	29.02
1.0789	20.49	1.0854	22.20	1.0919	23.91	1.0984	25.62	1.1049	27.33	1.1114	29.04
1.0790	20.52	1.0855	22.22	1.0920	23.93	1.0985	25.64	1.1050	27.35	1.1115	29.07
1.0791	20.55	1.0856	22.25	1.0921	23.96	1.0986	25.67	1.1051	27.38	1.1116	29.09
1.0792	20.57	1.0857	22.28	1.0922	23.99	1.0987	25.70	1.1052	27.41	1.1117	29.12
1.0793	20.60	1.0858	22.30	1.0923	24.01	1.0988	25.72	1.1053	27.43	1.1118	29.15
1.0794	20.62	1.0859	22.33	1.0924	24.04	1.0989	25.75	1.1054	27.46	1.1119	29.17
1.0795	20.65	1.0860	22.36	1.0925	24.07	1.0990	25.78	1.1055	27.49	1.1120	29.20
1.0796	20.68	1.0861	22.38	1.0926	24.09	1.0991	25.80	1.1056	27.51	1.1121	29.23
1.0797	20.70	1.0862	22.41	1.0927	24.12	1.0992	25.83	1.1057	27.54	1.1122	29.25
1.0798	20.73	1.0863	22.43	1.0928	24.14	1.0993	25.85	1.1058	27.57	1.1123	29.28
1.0799	20.75	1.0864	22.46	1.0929	24.17	1.0994	25.88	1.1059	27.59	1.1124	29.31
1.0800	20.78	1.0865	22.49	1.0930	24.20	1.0995	25.91	1.1060	27.62	1.1125	29.33
1.0801	20.81	1.0866	22.51	1.0931	24.22	1.0996	25.93	1.1061	27.65	1.1126	29.36
1.0802	20.83	1.0867	22.54	1.0932	24.25	1.0997	25.96	1.1062	27.67	1.1127	29.39
1.0803	20.86	1.0868	22.57	1.0933	24.27	1.0998	25.99	1.1063	27.70	1.1128	29.41
1.0804	20.89	1.0869	22.59	1.0934	24.30	1.0999	26.01	1.1064	27.72	1.1129	29.44
1.0805	20.91	1.0870	22.62	1.0935	24.33	1.1000	26.04	1.1065	27.75	1.1130	29.47
1.0806	20.94	1.0871	22.65	1.0936	24.35	1.1001	26.06	1.1066	27.78	1.1131	29.49
1.0807	20.96	1.0872	22.67	1.0937	24.38	1.1002	26.09	1.1067	27.80	1.1132	29.52
1.0808	20.99	1.0873	22.70	1.0938	24.41	1.1003	26.12	1.1068	27.83	1.1133	29.54
1.0809	21.02	1.0874	22.72	1.0939	24.43	1.1004	26.14	1.1069	27.86	1.1134	29.57
1.0810	21.04	1.0875	22.75	1.0940	24.46	1.1005	26.17	1.1070	27.88	1.1135	29.60
1.0811	21.07	1.0876	22.78	1.0941	24.49	1.1006	26.20	1.1071	27.96	1.1136	29.62
1.0812	21.10	1.0877	22.80	1.0942	24.51	1.1007	26.22	1.1072	27.93	1.1137	29.65
1.0813	21.12	1.0878	22.83	1.0943	24.54	1.1008	26.25	1.1073	27.96	1.1138	29.68
1.0814	21.15	1.0879	22.86	1.0944	24.57	1.1009	26.27	1.1074	27.99	1.1139	29.70
1.0815	21.17	1.0880	22.88	1.0945	24.59	1.1010	26.30	1.1075	28.01	1.1140	29.73
1.0816	21.20	1.0881	22.91	1.0946	24.62	1.1011	26.33	1.1076	28.04	1.1141	29.76
1.0817	21.23	1.0882	22.93	1.0947	24.64	1.1012	26.35	1.1077	28.07	1.1142	29.78
1.0818	21.25	1.0883	22.96	1.0948	24.67	1.1013	26.38	1.1078	28.09	1.1143	29.81
1.0819	21.28	1.0884	22.99	1.0949	24.70	1.1014	26.41	1.1079	28.12	1.1144	29.83
1.0820	21.31	1.0885	23.01	1.0950	24.72	1.1015	26.43	1.1080	28.15	1.1145	29.86
1.0821	21.33	1.0886	23.04	1.0951	24.75	1.1016	26.46	1.1081	28.17	1.1146	29.89
1.0822	21.36	1.0887	23.07	1.0952	24.78	1.1017	26.49	1.1082	28.20	1.1147	29.91
1.0823	21.38	1.0888	23.09	1.0953	24.80	1.1018	26.51	1.1083	28.22	1.1148	29.94
1.0824	21.41	1.0889	23.12	1.0954	24.83	1.1019	26.54	1.1084	28.25	1.1149	29.96
1.0825	21.44	1.0890	23.14	1.0955	24.85	1.1020	26.56	1.1085	28.28	1.1150	29.99
1.0826	21.46	1.0891	23.17	1.0956	24.88	1.1021	26.59	1.1086	28.30	1.1151	30.02
1.0827	21.49	1.0892	23.20	1.0957	24.91	1.1022	26.62	1.1087	28.33	1.1152	30.04
1.0828	21.52	1.0893	23.22	1.0958	24.93	1.1023	26.64	1.1088	28.36	1.1153	30.7
1.0829	21.54	1.0894	23.25	1.0959	24.96	1.1024	26.67	1.1089	28.38	1.1154	30.10
1.0830	21.57	1.0895	23.28	1.0960	24.99	1.1025	26.70	1.1090	28.41	1.1155	30.13
1.0831	21.59	1.0896	23.31	1.0961	25.01	1.1026	26.72	1.1091	28.43	1.1156	30.15
1.0832	21.62	1.0897	23.33	1.0962	25.04	1.1027	26.75	1.1092	28.46	1.1157	30.18
1.0833	21.65	1.0898	23.36	1.0963	25.07	1.1028	26.78	1.1093	28.49	1.1158	30.21
1.0834	21.67	1.0899	23.38	1.0964	25.09	1.1029	26.80	1.1094	28.51	1.1159	30.23
1.0835	21.70	1.0900	23.41	1.0965	25.12	1.1030	26.83	1.1095	28.54		
1.0836	21.73	1.0901	23.43	1.0966	25.14	1.1031	26.85	1.1096	28.57		
1.0837	21.75	1.0902	23.46	1.0967	25.17	1.1032	26.88	1.1097	28.59		
1.0838	21.78	1.0903	23.49	1.0968	25.20	1.1033	26.91	1.1098	28.62		
1.0839	21.80	1.0904	23.51	1.0969	25.22	1.1034	26.93	1.1099	28.65		
1.0840	21.83	1.0905	23.54	1.0970	25.25	1.1035	26.96	1.1100	28.67		
1.0841	21.86	1.0906	23.57	1.0971	25.28	1.1036	26.99	1.1101	28.70		
1.0842	21.88	1.0907	23.59	1.0972	25.30	1.1037	27.01	1.1102	28.73		
1.0843	21.91	1.0908	23.62	1.0673	25.33	1.1038	27.04	1.1103	28.75		
1.0844	21.94	1.0909	23.65	1.0974	25.36	1.1039	27.07	1.1104	28.78		

as an indicator. Each cubic centimeter of tenth-normal alkali is equivalent to 0.006 gram acetic acid.

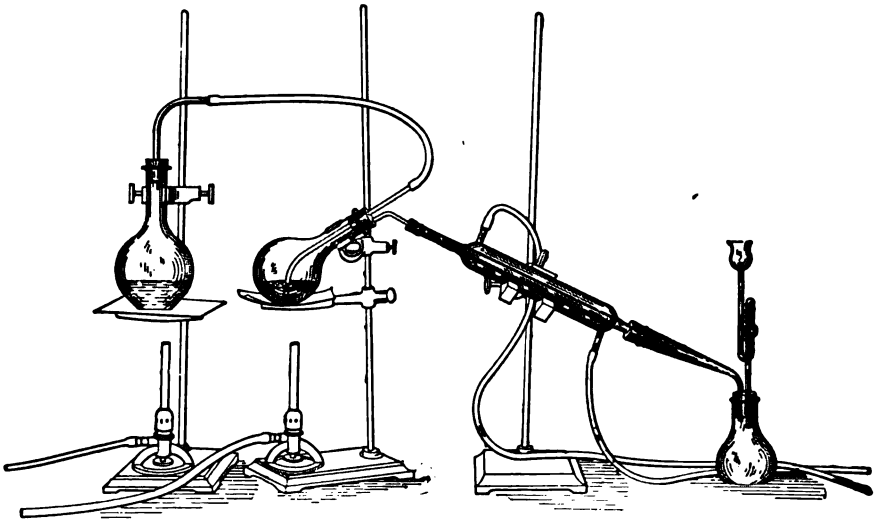


FIG. 114.—Apparatus for Determining Volatile Acids in Wine.

Non-volatile Acids.—These may be determined by difference, calculating the volatile acids for purposes of subtraction in terms of tartaric or other acid in which the total acidity is expressed. Non-volatile acid may be directly determined by evaporating to dryness a measured portion of the liquor, boiling the residue with water, and titrating the solution with the standard alkali.

Detection of Free Tartaric Acid.—*Nessler's Method.*—Some powdered cream of tartar is added to a portion of the wine in a corked flask,

Let stand at least fifteen hours at room temperature; decant the liquid from the separated acid potassium tartrate as rapidly as possible (using vacuum) through a Gooch crucible prepared with a very thin film of asbestos, transferring no more of the precipitate to the crucible than necessary. Wash the precipitate and filter three times with a small amount of a mixture of 15 grams potassium chloride, 20 cc. of 95% alcohol (specific gravity 0.81), and 100 cc. water, using not more than 20 cc. of the wash solution in all. Transfer the asbestos film and precipitate to the beaker in which the precipitation took place, wash out the Gooch crucible with hot water, add about 50 cc. of hot water, heat to boiling, and titrate the hot solution with decinormal sodium hydroxide, using delicate litmus tincture or litmus paper as indicator. Increase the number of cubic centimeters of decinormal alkali employed by 1.5 on account of the solubility of the precipitate. This figure multiplied by 0.015 gives the amount of total tartaric acid in grams per 100 cc.

Cream of Tartar.—Ignite the residue obtained from the evaporation of 50 cc. of wine as directed under the determination of ash. Exhaust the ash with hot water, add to the filtrate 25 cc. of decinormal hydrochloric acid, heat to incipient boiling, and titrate with decinormal alkali solution, using litmus as indicator. Deduct from 25 cc. the number of cubic centimeters of decinormal alkali employed, and multiply the remainder by 0.0188 for potassium bitartrate expressed in grams.

Free Tartaric Acid.—Add 25 cc. of decinormal hydrochloric acid to the ash of 50 cc. of wine, heat to incipient boiling, and titrate with decinormal sodium hydroxide, using litmus as indicator. Deduct the number of cubic centimeters of alkali employed from 25, and multiply the remainder by 0.0075 to obtain the amount of tartaric acid necessary to combine with all the ash (considering it to consist entirely of potash). Deduct the figure so obtained from the total tartaric acid for the free tartaric acid.

Determination of Free and Combined Malic Acid in Cider and Wine.—Evaporate 100 cc. of the sample on the water-bath to half its volume, cool, and treat first with 10 cc. of 10% calcium chloride solution, and then with ammonia to strong alkaline reaction. Let stand for an hour and filter. This removes the tartaric acid. Concentrate the filtrate by evaporation on the water-bath to 25 cc., add 75 cc. of 95% alcohol, heat to the boiling-point, and filter. Wash the residue with a mixture of 3 parts 95% alcohol and 1 part water, dry, and burn to an ash. Add 25 cc. of tenth-normal hydrochloric acid to the ash, dilute with water,

heat to boiling, and titrate with tenth-normal sodium hydroxide, using phenolphthalein as an indicator. Multiply the difference between 25 and the number of cubic centimeters required to neutralize by 0.0067 for the grams of malic acid.

Polarization.—Results are usually expressed in terms of the undiluted product. The simplest method consists in treating a measured amount of wine or cider with one-tenth of its volume of lead subacetate, filtering, and polarizing the filtrate in the 200-mm. tube. The reading is increased by 10% for the true direct polarization. If the filtrate is deeply colored after clarification with subacetate, as in the case of some artificially colored wines, use a 100-mm. tube and multiply by 2 the reading, increased by 10%.

If the reducing sugars are also to be determined, one can use the same solution for both the polarization and the reducing sugars, as follows:

Exactly neutralize with sodium hydroxide solution a measured quantity of the wine, using litmus paper as an indicator, and evaporate on the water-bath to about one-fourth its original volume. Wash with water, into the measuring-glass, add enough subacetate of lead solution to clarify, and make up with water to the original volume. Filter, and to a measured amount of the filtrate add 10% of its volume of a saturated solution of sodium sulphate. Again filter and submit to polarization a portion of the filtrate, making the 10% correction on the reading.

If the invert reading is desired, subject a portion of the filtrate to inversion as described under Sugars.

Determination of Reducing Sugar.—Determine the reducing sugar as dextrose by the Defren or the Allihn method. For this purpose dilute a portion of the wine, dealcoholized and clarified as described in the preceding section, so that it contains about one-half of 1% of sugar for the Defren and about 1% of sugar for the Allihn method. One may assume

the residue in 10 cc. of absolute alcohol, and transfer to a flask with 15 cc. of ether. Tightly cork the flask, shake, and allow to stand for some time. Then filter into a tared dish, wash with a mixture of absolute alcohol (1 part) and ether (1.5 parts), evaporate the filtrate to syrupy consistency on the water-bath, dry in a water-oven for one hour, and weigh as glycerin.

With plastered wines the results are too high, for the reason that the potassium sulphate present is decomposed by the lime to form potassium hydroxide, soluble in glycerin and alcohol. In such wines the above residue should be ignited, and the ash deducted from the first weight.

Determination of Potassium Sulphate.—Acidify 100 cc. of the sample with hydrochloric acid, heat to boiling, and add an excess of barium chloride solution. Filter, wash, dry, ignite, and weigh as barium sulphate, calculating the equivalent of potassium sulphate. The presence of the latter in excess of 0.06 gram per 100 cc. indicates plastering.

Determination of Tannin.—An approximate method of determining tannin is that of Nessler and Barth. 12 cc. of wine are treated with 30 cc. of alcohol and filtered. 35 cc. of the filtrate, which corresponds to 10 cc. of the wine, is evaporated to about 6 cc. and transferred to a 10-cc. graduated centrifuge tube. A few drops of 40% sodium acetate are then added, and a slight excess of 10% ferric chloride. The tube is corked, gently agitated, and allowed to stand for twenty-four hours. The volume of the precipitate is then measured, each cubic centimeter being equivalent to 0.033% of tannin in the wine.

Foreign Coloring Matters in Wine.—A wide variety of artificial colors have been found in red wine. Those most commonly employed at present are cochineal, fuchsin, and acid fuchsin.

The Pharmacopœia prescribes the following color tests:

If 2 cc. of red wine be mixed in a test-tube with 2 drops of chloroform and 4 cc. of normal potassium hydroxide, and the mixture carefully heated, the disagreeable odor of isonitril should not become preceptible (absence of various anilin colors).

If 50 cc. of red wine be treated with a slight excess of ammonia water, the liquid should acquire a green or brownish-green color; if it be then well shaken with 25 cc. of ether, the greater portion of the ethereal layer removed and evaporated in a porcelain capsule with an excess of acetic acid and a few fibers of uncolored silk, the latter should not acquire a crimson or violet color (absence of fuchsin).

If 25 cc. of red wine heated to about 45° C. be well agitated with 25 grams of manganese dioxide, the liquid filtered off and acidulated with

hydrochloric acid, it should not acquire a red color (absence of sulphofuchsin).

*Dupré's Method of Detection.**—Small cubes of jelly measuring about 2 cm. in thickness are prepared as follows: Dissolve 1 part of pure gelatin in 10 parts boiling water and pour upon a plate to harden. This is then cut into cubes of the above size by a sharp knife. When a wine is suspected of containing foreign color, one of the cubes is immersed therein and allowed to remain for twenty-four hours, after which it is removed, washed slightly in cold water, and cut through with a knife. If the color is a natural one, it will lightly tinge the outer surface of the cube, but will not permeate far below the surface, so that the inner portion of the cross-section will be largely free from color. Nearly all foreign coloring matters used in wine, such as most coal-tar dyes, cochineal, Brazil wood, logwood, etc., will be found to deeply permeate the jelly cube often to the center. Information as to the nature of the color may sometimes be gained by immersing the dyed jelly cube in weak ammonia. If the color be rosanilin, the cube is decolorized, if cochineal, a purple coloration will result, and if logwood, a brown tinge.

Cazeneuve's Methods for Detecting Colors in Wine.—While by no means complete, the following method of Cazeneuve as condensed and arranged by Gautier (*La Sophistication des Vins*) will often be found helpful. If other colors than these are evidently present, tests should be made as indicated in Chapter XVI. Cazeneuve employs the following reagents:

(1) Yellow oxide of mercury, finely pulverized.

(2) Lead hydrate, freshly precipitated, well washed, suspended in about twice its volume of water; to be kept in a stoppered bottle; should be renewed after several days' use.



To 10 cc. of the wine are added 0.2 gram finely powdered yellow oxide of mercury. Boil and pour upon a double filter.

- **Filtrate colorless after acidifying.**
  - Ten cc. of wine are treated with 10 grams of iron hydrate and brought to boiling.
    - Filtrate colorless ....
      - **Ten cc. wine are treated with 20 grams stannous hydrate and brought to boiling.**
        - Colorless. ... — *Pure wine*
        - Colored. ..... — *Wine colored with vegetable pigments*
      - **A fluorescent rose** ..... *Cochineal*
      - **A non-fluorescent rose** ..... *Eosin*
    - Filtrate colored .... — *Erythrosin*
- **Filtrate colored either before or after acidifying.**
  - Filtrate colorless.
    - The preceding solution is decolorized by ammonia.
      - **To a portion of the wine is added its weight of manganese dioxide. Acidify and filter.**
        - Filtrate colorless or yellowish. — *Fuchsin*
        - Filtrate yellow-red or red. — *Acid fuchsin*
      - The preceding solution is not changed by ammonia. Acidify and boil up with wool fibers. Wash the fibers, dry, and treat them with pure concentrated H_2SO_4, which colors them as follows:
        - **Purple-violet** ..... *Roccellin*
        - **Blue-violet** ..... *Purple-red*
        - **Blue** ..... *Bordeaux red*
        - **Crimson** ..... *Ponceaux*
        - **Deep green** ..... *Biebrich scarlet*
        - **Indigo-blue** ..... *Croceine 3B*
        - **Violet** ..... *Croceine 7B*
  - **Filtrate is colored red** ..... *Safranin*
- **Filtrate colored red.** 0 cc. of the wine are treated with 2 grams lead hydrate and filtered.
  - **Fuchsin red** ..... — *Tropeolin* 000, 1 and 2
  - **Orange-brown** ..... — *Orange* 1 and 2
  - **Yellow-orange** ..... — *Tropeolin* 0
  - **Violet-red** ..... — *Tropeolin* y.
  - **Brownish yellow** ..... — *Tropeolin* oo (Orange IV)
  - **Brown-yellow** ..... *Helianthin*, Orange III
  - **Brown** ..... *Chrysoin*
  - **Yellow**, becoming salmon-red on diluting with water ..... *Resorcin*
  - **Blue-green** ..... *Solid yellow*
  - **Brown-yellow** ..... *Yellow* N
  - **Golden yellow** ..... *Yellow* NS
  - Ammonia is added to the wine freed from its natural color by yellow oxide of mercury. Causes a blue to yellow precipitate. — *Martius yellow*
- **Filtrate colored yellow.** 10 cc. of the wine are warmed with 2 grams lead hydrate. Filter.
  - Filtrate colored yellow. A large excess of lead hydrate is added and the liquid is boiled.
    - Solution colorless. Wool fibers are dyed, washed, dried, and treated with pure concentrated H_2SO_4. Color produced is
      - Solution colored yellow, but only faintly. Wool fibers are dyed, washed, dried, and treated with pure concentrated H_2SO_4. Color produced is
        - **Filtrate is colored red. Boil up with wool fibers, which after washing and drying are treated with pure concentrated H_2SO_4. Color produced is**
          - **Fuchsin red** ..... — *Tropeolin* 000, 1 and 2
          - **Orange-brown** ..... — *Orange* 1 and 2
          - **Yellow-orange** ..... — *Tropeolin* 0
          - **Violet-red** ..... — *Tropeolin* y.
          - **Brownish yellow** ..... — *Tropeolin* oo (Orange IV)
          - **Brown-yellow** ..... *Helianthin*, Orange III
          - **Brown** ..... *Chrysoin*
          - **Yellow**, becoming salmon-red on diluting with water ..... *Resorcin*
          - **Blue-green** ..... *Solid yellow*
          - **Brown-yellow** ..... *Yellow* NS
          - **Golden yellow** ..... *Martius yellow*
    - **Filtrate colored blue. The wine is boiled with collodion silk.** — **Fiber is colored blue**

MALT LIQUORS. BEER.

In its widest sense beer may be defined as the product of fermentation of an infusion of almost any farinaceous grain with various bitter extractives, but unless otherwise qualified it should be strictly applied to the beverage resulting from the fermentation of malted barley and hops. In the manufacture of beer two distinct processes are employed, viz., malting or sprouting the grain, and brewing. Many brewers do nothing but the latter, buying their malt already prepared.

Malting.—For the preparation of malt, the barley is steeped in water for several days, after which the water is drained off and the moist grain is "couched," or piled in heaps, on a cement floor, where it undergoes a spontaneous heating process, during which it germinates, forming the ferment diastase. When the maximum amount of diastase has been produced, indicated by the length of growth of the sprout, or "acrospire" within the grain, the germination is checked by spreading the grain in layers over a perforated iron floor, and finally subjecting it to artificial heat. The character of the malt and of the beer produced from it depends largely on the heat at which the "green" malt is kiln dried. If dried between 32° and 37° C. it forms pale malt, which produces the lightest grades of beer. Most beer is made from malt dried at higher temperatures, say from 38° to 50°, the depth of color of the liquor varying with the heat to which the malt has been subjected, while the color of the malt varies from the "pale" through the "amber" to "brown," or even black. The darkest grades are sometimes dried at temperatures over 100° C., even to the point where the starch becomes caramelized.

A more modern method consists in the so-called pneumatic malting



Brewing.—The malt, or mixture of malt and raw grain, is first crushed and "mashed" by stirring with water in tubs at 50° to 60° C., finally heating to 70°. During this process the conversion of the starch to maltose and dextrin takes place. The resulting liquor is known as "wort," containing, besides maltose and dextrin, peptones and amides. The clear wort is then drawn off from the residue, and boiled to concentrate the product and to sterilize it, after which hops (the female flower of the *Humulus lupulus*) are added and the boiling continued. Hops contain resins, bitter principles, tannic acid, and a peculiar essential oil, all of which are to some extent imparted to the wort. After cooling and settling, the clear wort is run into fermenting-vats, where selected yeast, usually *saccharomyces cerevisiæ*, is added, and the alcoholic fermentation allowed to proceed. The temperature greatly affects the character of the fermentation. If kept between 5° and 8° C., a slow fermentation proceeds, known as bottom fermentation, during which the yeast settles out at the bottom. This is much more easily controlled than the quick or top fermentation, which takes place at from 15° to 18°, much of the yeast in the latter case being carried to the surface, from which it is finally removed by skimming. In either case the yeast feeds upon the albuminous matter present. At the proper stage the beer is drawn off from the larger portion of the yeast, and run into casks, or tuns, in which an after-fermentation proceeds. The beer is finally clarified by treatment with gelatin or beech shavings or chips, to which the floating yeast-cells and other impurities attach themselves. It is finally stored in barrels coated with brewers' pitch, or pasteurized at 60° C. and bottled.

Varieties of Beer.—Formerly the division of beers into "lager," "schenk," and "bock" was made by reason of the fact that beer had to be brewed under certain climatic conditions and at certain seasons only. Now, with improved means for artificial refrigeration, and with better methods controlling all stages of the process, these distinctions are less marked.

Lager Beer (from *lager*, a storehouse) is a term originally applied to Bavarian beer, but is now given to any beer that has been stored several months. Formerly lager beer was made early in the winter, and stored in cool cellars till the following spring or summer, during nearly all of which time a slow after-fermentation took place. The best lager beers contain a low proportion of hops, and are high in extract and alcohol.

Schenk Beer is a quickly fermented beer made in winter for immedi-

ate use. It is brewed in from four to six weeks and will not keep long without souring.

Bock Beer, according to older systems of nomenclature, occupied a middle place between lager and schenk, being an extra strong beer brewed for spring use and made in limited quantities, not being intended for storage.

Berlin Weiss Bier is prepared by the quick or top fermentation of a wort consisting of a mixture of malted barley and wheat with hops. It is high in carbon dioxide, being usually bottled before the second fermentation has ended.

Ale is virtually the English name for beer. It is usually lighter colored than lager beer, being made from pale malt by quick or top fermentation, and containing rather more hops than beer. It has a high content of sugar, due to checking fermentation at an earlier stage than in ordinary beer.

Porter is a dark ale, the deep color of which should be due to the use of brown malt dried at a high temperature, but which is sometimes colored by the admixture of caramel. It has a large extract, chiefly sugar.

Stout is an extra-strong porter, being high both in alcohol and extract.

Composition of Beer.—Beer is a somewhat complex liquor. Besides water, alcohol, and sugar, it contains carbon dioxide, succinic acid, dextrin, glycerin, tannic acid, the resinous bitter principles of hops, nitrogenous bodies (chiefly peptones and amides), alkaline and lime salts (chiefly phosphates), fat (traces), acetic acid and lactic acid. The latter acid constitutes the chief fixed acid of beer.

The following analyses of different varieties of beer are due to König:



of liquor sold every day to patrons by the bottle or glass, were analyzed by the Board of Health with the following results:

		Per Cent of Original Wort Extract.	Per Cent of Alcohol by Weight.	Per Cent of Extract.
Beer—	Maximum	18.91	7.07	7.76
	Minimum	7.33	1.10	3.67
	Mean	15.04	4.45	5.92
Pale ale—	Maximum	15.99	5.37	5.47
	Minimum	10.95	3.53	3.38
	Mean	13.56	4.49	4.54

Five out of the 15 beer samples and 3 out of the 7 ale samples contained salicylic acid.

The percentage composition of the ash of German beer is thus given by König as the mean of 19 analyses:

Ash in 100 Parts Beer.	Potash.	Soda.	Lime.	Magnesia.	Iron Oxide.	Phosphoric Acid.	Sulphuric Acid.	Silica.	Chlorine.
0.306	33.67	8.94	2.78	6.24	0.48	31.35	3.47	9.29	2.93

Malt and Hop Substitutes.—By reason of the fluctuation in market price of these two chief constituents of beer, it sometimes becomes a question of economy to employ cheaper substitutes wholly or in part for one or the other. There are two classes of malt substitutes, (1) those which, like corn, rice, and wheat, are mixed directly with the malt before "mashing," and, like the malt, have to undergo a saccharous fermentation before being acted on by yeast, and (2) such substances as cane sugar, invert sugar, commercial glucose, and dextrin, which are added to the wort at a later stage in the brewing, just before the addition of the yeast, being in condition to be readily acted on by the latter.

Glucose is by far the most common malt substitute, by reason of the fact that its sugars much resemble those of malt, and are in readily fermentable form. Diastase forms from the malt dextrin and maltose, while commercial glucose contains dextrin, maltose, and dextrose.

When the price of malt is abnormally high, the addition of glucose is decidedly economical, but when ordinary conditions prevail, the cost of the two, figured with reference to their yield in alcohol and extract, is about the same. Aside from the question of economy, however, there are advantages in the use of glucose, such as diminishing the nitrogenous content of the wort without lessening the alcohol or extract yielded.

The nitrogenous matter left after fermentation is one of the chief causes of cloudiness or turbidity in the finished product, and is sometimes difficult to remove. By the use of glucose, especially in brewing clear bottled ales and sparkling pale beers, the appearance of the product is much enhanced. The temptation at times to add more glucose than is necessary to accomplish this is great. A high-grade malt may have as much as 40% of glucose added to its wort and still produce an acceptable beer. With a low-grade malt, glucose yields a very poor quality of beer. Hence the use of glucose may or may not be desirable, though it can hardly be considered unqualifiedly as an adulterant.

As to the employment of hop substitutes, the question of relative price again enters in. Only when the price of hops is high is there any special inducement to use substitutes. Quassia wood, chiretta, gentian, and calumba, all of which yield bitter principles, have been used in beer, and cannot be considered detrimental to health. Allen and Chattaway have found the first two in beer examined by them.* Such poisonous substances as cocculus indicus, picric acid, and strychnine are alleged to have been used as hop substitutes, but there is no authentic record of any of them having been found in recent years, if at all.

Adulteration of Beer and Standards of Purity.—The Committee on Standards of the A. O. A. C. in their proposed definitions and standards have distinguished between *malt beer*, which should be a malt liquor obtained by fermenting an infusion of malted barley, and adding thereto nothing but yeast and hops, and *beer*, or *lager beer*, which may contain non-injurious malt and hop substitutes in addition to malt. Their proposed standard for malt beer is as follows:

Alcohol should be not less than 4% by volume; the extract should



"malt liquors," so that when convictions are obtained, it is necessary for the analyst to distinguish between liquors brewed wholly or in part from malt and those in which no malt has been used, but which were brewed entirely from malt substitutes. This distinction is not always easy to make with precision. In the absence of malt, glucose is usually the sole source of alcohol in these beverages. Parsons * has shown that the most striking points of difference between malted and non-malted liquors are in their per cent of phosphoric acid and albuminoids, and that pure malt beer or ale should contain at least 0.04% P_2O_5, and 0.25% albuminoids (N×6.25). A low ash and high content of sulphates in the ash are also indicative of glucose. The following analyses made by Parsons clearly show these distinctions:

COMPOSITION OF SEVENTY-SIX SAMPLES OF AMERICAN MALT LIQUORS.

	Specific Gravity.	Alcohol by Volume.	Extract.	Albuminoids (N×6.25)	Phosphoric Acid.	Ash.	Sulphates in Ash.	Free Acid.
Average.....	1.0100	5.61	4.61	0.470	0.061	0.209	6.34	0.26
Maximum....	1.0210	7.85	7.64	0.614	0.095	0.296	12.67	0.87
Minimum. ...	1.0047	0.35	3.15	0.290	0.045	0.147	2.44	0.10

TYPICAL ANALYSES OF BEERS APPARENTLY NOT BREWED FROM MALT.

Number.	Specific Gravity.	Alcohol by Volume.	Extract.	Albuminoids (N×6.25)	Phosphoric Acid.	Ash.	Sulphates.	Free Acid.
1.....	1.0074	1.68	2.52	0.114	0.010	0.19	21.22	Normal
2.....	1.0098	2.63	3.40	0.215	0.023	0.180	.....	"
3.....	1.0062	2.27	2.25	0.150	0.015	0.124	11.30	"
4.....	1.0112	2.11	3.53	0.133	0.015	0.140	10.81	"
5.....	1.0041	1.85	1.73	0.031	0.010	0.088	12.50	"

The ash of the fifth sample is thus compared with that of the average beer as given by Blyth:

	Malt Beer (Blyth).	"No-malt" Beer (Parsons).
K_2O.....	37.22	12.93
Na_2O.....	8.04	19.61
CaO.....	1.93	Undetermined
MgO.....	5.51	"
Fe_2O_3.....	Trace	"
SO_3.....	1.44	10.81
P_2O_5.....	32.09	10.71
Cl.....	2.91	21.76
SiO_2.....	10.82	7.50

* Jour. Am. Chem. Soc., 24, 1902, p. 1170.

Preservatives in Beer.—Antiseptics are frequently added to malt liquors, salicylic acid being most commonly used. Fluorides of ammonium and sodium have been found in American beer. Other preservatives to be looked for are benzoic acid and sulphites. Beer casks are frequently "sulphured" or fumed with a solution of calcium bisulphite, so that the beer may derive its content of sulphites from this source.

In cases of police seizure of beer sold in bulk or in opened bottles for the purpose of ascertaining whether or not their alcoholic content exceeds certain limits fixed by law, a little formalin had best be added as soon possible after the seizure to prevent further fermentation. This is especially desirable in cases where there is likely to be some delay in making the analysis, so as to forestall any claim on the part of the defendant of additional alcohol being formed after the seizure. From 6 to 8 drops of a 40% solution of formaldehyde to a quart of beer is sufficient, and this quantity will not appreciably affect the analysis.

Arsenic in Beer.—In 1900 a very disastrous epidemic of arsenical poisoning occurred in Manchester, England, involving several thousand cases, many of which were fatal. The arsenic was traced to sulphuric acid, which entered into the manufacture of commercial glucose used in the beer, the acid found so highly arsenical being made from a certain variety of Swedish pyrites, which was found to be abnormally high in arsenic. There appeared to be no doubt whatever that the beer was the sole cause of the trouble. While the presence of arsenic was in this case accidental, carelessness was shown on the part of those having to do with the purity of the materials entering into the composition of the beer. Fortunately no other instances are on record of arsenical poisoning from malted liquors. A large number of samples of American beer have been



position of many of these beverages is identical with that of lager beer, excepting in alcoholic content, being made by substantially the same process and out of the same ingredients, but with the alcohol finally removed by steaming, so that the liquor comes within the limits of a temperance beverage.

Of this class is *Uno beer*, which ranges from 0.6 to 0.9 per cent in alcohol.

Methods of Analysis.—For determinations of specific gravity, alcohol, extract (by direct method), and ash see pp. 527, 528, and 547. For preservatives see Chapter XVII.

Calculation of Extract in Beer.—When extreme accuracy is desired, the result obtained by evaporating at 100° a weighed amount of the beer cannot be accepted, on account of the dehydration of the maltose at a temperature exceeding 75° C. Unless the evaporation is conducted at that temperature (a difficult operation), a closer approximation to the truth is obtained, especially with beer high in sugar, by calculation as follows.

Evaporate a measured quantity of the beer to one-fourth its volume on the water-bath, make up with water to its original measure, and determine the specific gravity of the dealcoholized beer. Then by means of Schultz and Ostermann's table, pp. 582–6, calculate the extract corresponding.

Original Gravity of Beer Wort and its Determination.—Following a long-established custom of the English excise, the duty on beer has been based on the specific gravity of the original wort, by which is meant the wort of the beer before any of its sugar has been lost by fermentation.

From the content of alcohol in the beer the sugar originally present in the wort may be calculated, assuming that the alcohol amounts to about half the sugar used up in fermentation.

Obtain the specific gravity of the beer, dealcoholized and made up to its original volume, as in the calculation of the extract. This is called the "extract gravity." Note the specific gravity corresponding to the alcohol found, i.e., the specific gravity of the distillate in the alcohol determination, when made up to the original volume, and subtract this from 1. The difference is known arbitrarily as the "degree of spirit indication."

From the table of Graham, Hofmann, and Redwood,* p. 587, the "degrees of gravity lost" corresponding to the "spirit indication"

* Report on Original Gravities, 1852; Allen's Com. Org. Anal., I., p. 136.

EXTRACT IN BEER WORT.*

[ACCORDING TO SCHULTZ AND OSTERMANN.]

Specific Gravity at 15° C.	Extract. Per Cent by Weight	Extract. Grams per 100 cc.	Specific Gravity at 15° C.	Extract. Per Cent by Weight	Extract. Grams per 100 cc.	Specific Gravity at 15° C.	Extract. Per Cent by Weight	Extract. Grams per 100 cc.	Specific Gravity at 15° C.	Extract. Per Cent by Weight	Extract. Grams per 100 cc.
1.0000	0.00	0.00	1.0065	1.69	1.70	1.0130	3.35	3.39	1.0195	5.06	5.16
1.0001	0.03	0.03	1.0066	1.72	1.73	1.0131	3.38	3.42	1.0196	5.09	5.19
1.0002	0.05	0.05	1.0067	1.74	1.75	1.0132	3.41	3.46	1.0197	5.12	5.22
1.0003	0.08	0.08	1.0068	1.77	1.78	1.0133	3.43	3.48	1.0198	5.15	5.25
1.0004	0.10	0.10	1.0069	1.79	1.80	1.0134	3.46	3.51	1.0199	5.17	5.27
1.0005	0.13	0.13	1.0070	1.82	1.83	1.0135	3.48	3.53	1.0200	5.20	5.30
1.0006	0.16	0.16	1.0071	1.84	1.85	1.0136	3.51	3.56	1.0201	5.23	5.34
1.0007	0.18	0.18	1.0072	1.87	1.88	1.0137	3.54	3.59	1.0202	5.25	5.36
1.0008	0.21	0.21	1.0073	1.90	1.91	1.0138	3.56	3.61	1.0203	5.28	5.39
1.0009	0.24	0.24	1.0074	1.92	1.93	1.0139	3.59	3.64	1.0204	5.30	5.41
1.0010	0.26	0.26	1.0075	1.95	1.96	1.0140	3.61	3.66	1.0205	5.33	5.44
1.0011	0.29	0.29	1.0076	1.97	1.98	1.0141	3.64	3.69	1.0206	5.35	5.46
1.0012	0.31	0.31	1.0077	2.00	2.02	1.0142	3.66	3.71	1.0207	5.38	5.49
1.0013	0.34	0.34	1.0078	2.02	2.04	1.0143	3.69	3.74	1.0208	5.40	5.51
1.0014	0.37	0.37	1.0079	2.05	2.07	1.0144	3.72	3.77	1.0209	5.43	5.54
1.0015	0.39	0.39	1.0080	2.07	2.09	1.0145	3.74	3.79	1.0210	5.45	5.56
1.0016	0.42	0.42	1.0081	2.10	2.12	1.0146	3.77	3.83	1.0211	5.48	5.60
1.0017	0.45	0.45	1.0082	2.12	2.14	1.0147	3.79	3.85	1.0212	5.50	5.62
1.0018	0.47	0.47	1.0083	2.15	2.17	1.0148	3.82	3.88	1.0213	5.53	5.65
1.0019	0.50	0.50	1.0084	2.17	2.19	1.0149	3.85	3.91	1.0214	5.55	5.67
1.0020	0.52	0.52	1.0085	2.20	2.22	1.0150	3.87	3.93	1.0215	5.57	5.69
1.0021	0.55	0.55	1.0086	2.23	2.25	1.0151	3.90	3.96	1.0216	5.60	5.72
1.0022	0.58	0.58	1.0087	2.25	2.27	1.0152	3.92	3.98	1.0217	5.62	5.74
1.0023	0.60	0.60	1.0088	2.28	2.30	1.0153	3.95	4.01	1.0218	5.65	5.77
1.0024	0.63	0.63	1.0089	2.30	2.32	1.0154	3.97	4.03	1.0219	5.67	5.79
1.0025	0.66	0.66	1.0090	2.33	2.35	1.0155	4.00	4.06	1.0220	5.70	5.83
1.0026	0.68	0.68	1.0091	2.35	2.37	1.0156	4.03	4.09	1.0221	5.72	5.85
1.0027	0.71	0.71	1.0092	2.38	2.40	1.0157	4.05	4.11	1.0222	5.75	5.88
1.0028	0.73	0.73	1.0093	2.41	2.43	1.0158	4.08	4.14	1.0223	5.77	5.90
1.0029	0.76	0.76	1.0094	2.43	2.45	1.0159	4.10	4.17	1.0224	5.80	5.93
1.0030	0.79	0.79	1.0095	2.46	2.48	1.0160	4.13	4.20	1.0225	5.82	5.95
1.0031	0.81	0.81	1.0096	2.48	2.50	1.0161	4.16	4.23	1.0226	5.84	5.97
1.0032	0.84	0.84	1.0097	2.51	2.53	1.0162	4.18	4.25	1.0227	5.87	6.00
1.0033	0.87	0.87	1.0098	2.53	2.55	1.0163	4.21	4.28	1.0228	5.89	6.02
1.0034	0.89	0.89	1.0099	2.56	2.59	1.0164	4.23	4.30	1.0229	5.92	6.06
1.0035	0.92	0.92	1.0100	2.58	2.61	1.0165	4.26	4.33	1.0230	5.94	6.08
1.0036	0.94	0.94	1.0101	2.61	2.64	1.0166	4.28	4.35	1.0231	5.97	6.11
1.0037	0.97	0.97	1.0102	2.64	2.67	1.0167	4.31	4.38	1.0232	5.99	6.13
1.0038	1.00	1.00	1.0103	2.66	2.69	1.0168	4.34	4.41	1.0233	6.02	6.16

EXTRACT IN BEER WORT—(*Continued*).

Specific Gravity at 15° C.	Extract. Per Cent by Weight	Extract. Grams per 100 cc.	Specific Gravity at 15° C.	Extract. Per Cent by Weight	Extract. Grams per 100 cc.	Specific Gravity at 15° C.	Extract. Per Cent by Weight	Extract. Grams per 100 cc.	Specific Gravity at 15° C.	Extract. Per Cent by Weight	Extract. Grams per 100 cc.
1.0260	6.71	6.88	1.0325	8.27	8.54	1.0390	9.92	10.31	1.0455	11.53	12.05
1.0261	6.74	6.92	1.0326	8.29	8.56	1.0391	9.95	10.34	1.0456	11.55	12.08
1.0262	6.77	6.95	1.0327	8.32	8.59	1.0392	9.97	10.36	1.0457	11.57	12.10
1.0263	6.80	6.98	1.0328	8.34	8.61	1.0393	9.99	10.38	1.0458	11.60	12.13
1.0264	6.82	7.00	1.0329	8.37	8.65	1.0394	10.02	10.41	1.0459	11.62	12.15
1.0265	6.85	7.03	1.0330	8.40	8.68	1.0395	10.04	10.44	1.0460	11.65	12.19
1.0266	6.88	7.06	1.0331	8.43	8.71	1.0396	10.06	10.46	1.0461	11.67	12.21
1.0267	6.91	7.09	1.0332	8.45	8.73	1.0397	10.09	10.49	1.0462	11.70	12.24
1.0268	6.93	7.12	1.0333	8.48	8.76	1.0398	10.11	10.51	1.0463	11.72	12.26
1.0269	6.96	7.15	1.0334	8.51	8.79	1.0399	10.13	10.53	1.0464	11.75	12.30
1.0270	6.99	7.18	1.0335	8.53	8.82	1.0400	10.16	10.57	1.0465	11.77	12.32
1.0271	7.01	7.20	1.0336	8.56	8.85	1.0401	10.18	10.59	1.0466	11.79	12.34
1.0272	7.04	7.23	1.0337	8.59	8.88	1.0402	10.20	10.61	1.0467	11.82	12.37
1.0273	7.07	7.26	1.0338	8.61	8.90	1.0403	10.23	10.64	1.0468	11.84	12.39
1.0274	7.10	7.29	1.0339	8.64	8.93	1.0404	10.25	10.66	1.0469	11.87	12.43
1.0275	7.12	7.32	1.0340	8.67	8.96	1.0405	10.27	10.69	1.0470	11.89	12.45
1.0276	7.15	7.35	1.0341	8.70	9.00	1.0406	10.30	10.72	1.0471	11.92	12.48
1.0277	7.18	7.38	1.0342	8.72	9.02	1.0407	10.32	10.74	1.0472	11.94	12.50
1.0278	7.21	7.41	1.0343	8.75	9.05	1.0408	10.35	10.77	1.0473	11.97	12.54
1.0279	7.23	7.43	1.0344	8.78	9.08	1.0409	10.37	10.79	1.0474	11.99	12.56
1.0280	7.26	7.46	1.0345	8.80	9.10	1.0410	10.40	10.83	1.0475	12.01	12.58
1.0281	7.28	7.48	1.0346	8.83	9.14	1.0411	10.42	10.85	1.0476	12.04	12.61
1.0282	7.30	7.51	1.0347	8.86	9.17	1.0412	10.45	10.88	1.0477	12.06	12.64
1.0283	7.33	7.54	1.0348	8.88	9.19	1.0413	10.47	10.90	1.0478	12.09	12.67
1.0284	7.35	7.56	1.0349	8.91	9.22	1.0414	10.50	10.93	1.0479	12.11	12.69
1.0285	7.37	7.58	1.0350	8.94	9.25	1.0415	10.52	10.96	1.0480	12.14	12.72
1.0286	7.39	7.60	1.0351	8.97	9.28	1.0416	10.55	10.99	1.0481	12.16	12.74
1.0287	7.42	7.63	1.0352	8.99	9.31	1.0417	10.57	11.01	1.0482	12.19	12.78
1.0288	7.44	7.65	1.0353	9.02	9.34	1.0418	10.60	11.04	1.0483	12.21	12.80
1.0289	7.46	7.68	1.0354	9.05	9.37	1.0419	10.62	11.06	1.0484	12.23	12.82
1.0290	7.48	7.70	1.0355	9.07	9.39	1.0420	10.65	11.10	1.0485	12.26	12.85
1.0291	7.51	7.73	1.0356	9.10	9.42	1.0421	10.67	11.12	1.0486	12.28	12.88
1.0292	7.53	7.75	1.0357	9.13	9.46	1.0422	10.70	11.15	1.0487	12.31	12.91
1.0293	7.55	7.77	1.0358	9.15	9.48	1.0423	10.72	11.17	1.0488	12.33	12.93
1.0294	7.57	7.79	1.0359	9.18	9.51	1.0424	10.75	11.21	1.0489	12.36	12.96
1.0295	7.60	7.82	1.0360	9.21	9.54	1.0425	10.77	11.23	1.0490	12.38	12.99
1.0296	7.62	7.85	1.0361	9.24	9.57	1.0426	10.80	11.26	1.0491	12.41	13.02
1.0297	7.64	7.87	1.0362	9.26	9.60	1.0427	10.82	11.28	1.0492	12.43	13.04
1.0298	7.66	7.89	1.0363	9.29	9.63	1.0428	10.85	11.31	1.0493	12.45	13.06
1.0299	7.69	7.92	1.0364	9.31	9.65	1.0429	10.88	11.35	1.0494	12.48	13.10
1.0300	7.71	7.94	1.0365	9.34	9.68	1.0430	10.90	11.37	1.0495	12.50	13.12
1.0301	7.73	7.96	1.0366	9.37	9.70	1.0431	10.93	11.40	1.0496	12.53	13.15
1.0302	7.75	7.98	1.0367	9.38	9.72	1.0432	10.95	11.42	1.0497	12.55	13.17
1.0303	7.77	8.01	1.0368	9.41	9.76	1.0433	10.98	11.46	1.0498	12.58	13.21
1.0304	7.80	8.04	1.0369	9.43	9.78	1.0434	11.00	11.48	1.0499	12.60	13.23
1.0305	7.82	8.06	1.0370	9.46	9.81	1.0435	11.03	11.51	1.0500	12.63	13.26
1.0306	7.84	8.08	1.0371	9.48	9.83	1.0436	11.05	11.53	1.0501	12.65	13.28
1.0307	7.86	8.11	1.0372	9.50	9.85	1.0437	11.08	11.56	1.0502	12.67	13.30
1.0308	7.89	8.14	1.0373	9.53	9.88	1.0438	11.10	11.59	1.0503	12.70	13.34
1.0309	7.91	8.16	1.0374	9.55	9.91	1.0439	11.13	11.62	1.0504	12.72	13.36
1.0310	7.93	8.18	1.0375	9.57	9.93	1.0440	11.15	11.64	1.0505	12.75	13.39
1.0311	7.95	8.20	1.0376	9.59	9.95	1.0441	11.18	11.67	1.0506	12.77	13.42
1.0312	7.98	8.23	1.0377	9.62	9.98	1.0442	11.20	11.70	1.0507	12.80	13.45
1.0313	8.00	8.25	1.0378	9.64	10.00	1.0443	11.23	11.73	1.0508	12.82	13.47
1.0314	8.02	8.27	1.0379	9.66	10.03	1.0444	11.25	11.75	1.0509	12.85	13.50
1.0315	8.04	8.29	1.0380	9.69	10.06	1.0445	11.28	11.78	1.0510	12.87	13.53
1.0316	8.07	8.32	1.0381	9.71	10.08	1.0446	11.30	11.80	1.0511	12.90	13.56
1.0317	8.09	8.35	1.0382	9.73	10.10	1.0447	11.33	11.84	1.0512	12.92	13.58
1.0318	8.11	8.37	1.0383	9.76	10.14	1.0448	11.35	11.86	1.0513	12.94	13.60
1.0319	8.14	8.39	1.0384	9.78	10.16	1.0449	11.38	11.89	1.0514	12.97	13.64
1.0320	8.16	8.42	1.0385	9.81	10.19	1.0450	11.40	11.91	1.0515	12.99	13.66
1.0321	8.18	8.44	1.0386	9.83	10.21	1.0451	11.43	11.95	1.0516	13.02	13.69
1.0322	8.20	8.46	1.0387	9.85	10.23	1.0452	11.45	11.97	1.0517	13.04	13.71
1.0323	8.22	8.49	1.0388	9.88	10.26	1.0453	11.48	12.00	1.0518	13.07	13.75
1.0324	8.25	8.52	1.0389	9.90	10.29	1.0454	11.50	12.02	1.0519	13.09	13.77

EXTRACT IN BEER WORT—(*Continued*).

Specific Gravity at 15° C.	Extract. Per Cent by Weight	Extract. Grams per 100 cc.	Specific Gravity at 15° C.	Extract. Per Cent by Weight	Extract. Grams per 100 cc.	Specific Gravity at 15° C.	Extract. Per Cent by Weight	Extract. Grams per 100 cc.	Specific Gravity at 15° C.	Extract. Per Cent by Weight	Extract. Grams per 100 cc.
1.0520	13.12	13.80	1.0585	14.75	15.61	1.0650	16.25	17.31	1.0715	17.81	19.08
1.0521	13.14	13.82	1.0586	14.78	15.65	1.0651	16.27	17.33	1.0716	17.84	19.12
1.0522	13.16	13.85	1.0587	14.81	15.68	1.0652	16.30	17.36	1.0717	17.86	19.14
1.0523	13.19	13.88	1.0588	14.83	15.70	1.0653	16.32	17.39	1.0718	17.88	19.16
1.0524	13.21	13.90	1.0589	14.86	15.74	1.0654	16.35	17.42	1.0719	17.90	19.19
1.0525	13.24	13.94	1.0590	14.89	15.77	1.0655	16.37	17.44	1.0720	17.93	19.22
1.0526	13.26	13.96	1.0591	14.91	15.79	1.0656	16.40	17.48	1.0721	17.95	19.24
1.0527	13.29	13.99	1.0592	14.94	15.82	1.0657	16.42	17.50	1.0722	17.97	19.27
1.0528	13.31	14.01	1.0593	14.96	15.85	1.0658	16.45	17.53	1.0723	17.99	19.29
1.0529	13.34	14.05	1.0594	14.99	15.88	1.0659	16.47	17.56	1.0724	18.02	19.32
1.0530	13.36	14.07	1.0595	15.02	15.91	1.0660	16.50	17.59	1.0725	18.04	19.35
1.0531	13.38	14.09	1.0596	15.04	15.94	1.0661	16.52	17.61	1.0726	18.06	19.37
1.0532	13.41	14.12	1.0597	15.07	15.97	1.0662	16.54	17.63	1.0727	18.08	19.39
1.0533	13.43	14.15	1.0598	15.09	15.99	1.0663	16.57	17.67	1.0728	18.11	19.43
1.0534	13.46	14.18	1.0599	15.11	16.02	1.0664	16.59	17.69	1.0729	18.13	19.45
1.0535	13.48	14.20	1.0600	15.14	16.05	1.0665	16.62	17.73	1.0730	18.15	19.47
1.0536	13.51	14.23	1.0601	15.16	16.07	1.0666	16.64	17.75	1.0731	18.17	19.50
1.0537	13.53	14.26	1.0602	15.18	16.09	1.0667	16.67	17.78	1.0732	18.20	19.53
1.0538	13.56	14.29	1.0603	15.20	16.12	1.0668	16.69	17.80	1.0733	18.22	19.55
1.0539	13.58	14.31	1.0604	15.23	16 15	1 0669	16.72	17.84	1.0734	18.24	19.58
1.0540	13.61	14.34	1.0605	15.25	16.17	1.0670	16.74	17.86	1.0735	18.26	19.60
1.0541	13.63	14.37	1.0606	15.27	16.20	1.0671	16.76	17.88	1.0736	18.29	19.64
1.0542	13.66	14.40	1.0607	15.29	16.22	1.0672	16.79	17.92	1.0737	18.31	19.66
1.0543	13.68	14.42	1.0608	15.31	16.24	1.0673	16.81	17.94	1.0738	18.33	19.68
1.0544	13.71	14.46	1.0609	15.34	16.27	1.0674	16.84	17.98	1.0739	18.35	19.71
1.0545	13.73	14.48	1.0610	15.36	16.30	1.0675	16.86	18.00	1.0740	18.38	19.74
1.0546	13.76	14.51	1.0611	15.38	16.32	1.0676	16.89	18.03	1.0741	18.40	19.76
1.0547	13.78	14.53	1.0612	15.40	16.34	1.0677	16.91	18.05	1.0742	18.42	19.79
1.0548	13.81	14.57	1.0613	15.43	16.38	1.0678	16.94	18.09	1.0743	18.44	19.81
1.0549	13.83	14.59	1.0614	15.45	16.40	1.0679	16.96	18.11	1.0744	18.47	19.84
1.0550	13.86	14.62	1.0615	15.47	16.42	1.0680	16.99	18.15	1.0745	18.49	19.87
1.0551	13.88	14.64	1.0616	15.49	16.44	1.0681	17.01	18.17	1.0746	18.51	19.89
1.0552	13.91	14.68	1.0617	15.52	16.48	1.0682	17.03	18.19	1.0747	18.53	19.91
1.0553	13.93	14.70	1.0618	15.54	16.50	1.0683	17.06	18.23	1.0748	18.55	19.94
1.0554	13.96	14.73	1.0619	15.56	16.52	1.0684	17.08	18.25	1.0749	18.57	19.96
1.0555	13.98	14.76	1.0620	15.58	16.55	1.0685	17.11	18.28	1.0750	18.59	19.98
1.0556	14.01	14.79	1.0621	15.60	16.57	1.0686	17.13	18.31	1.0751	18.62	20.02
1.0557	14.03	14.81	1.0622	15.63	16.60	1.0687	17.16	18.34	1.0752	18.64	20.04
1.0558	14.06	14.84	1.0623	15.65	16.62	1.0688	17.18	18.36	1.0753	18.66	20.07
1.0559	14.08	14.87	1.0624	15.67	16.64	1.0689	17.21	18.40	1.0754	18.68	20.09

EXTRACT IN BEER WORT—(*Continued*).

Specific Gravity at 15° C.	Extract. Per Cent by Weight	Extract. Grams per 100 cc.	Specific Gravity at 15° C.	Extract. Per Cent by Weight	Extract. Grams per 100 cc.	Specific Gravity at 15° C.	Extract. Per Cent by Weight	Extract. Grams per 100 cc.	Specific Gravity at 15° C.	Extract. Per Cent by Weight	Extract. Grams per 100 cc.
1.0780	19.23	20.73	1.0845	20.70	22.45	1.0910	22.19	24.21	1.0975	23.59	25.89
1.0781	19.25	20.75	1.0846	20.73	22.48	1.0911	22.21	24.24	1.0976	23.61	25.92
1.0782	19.27	20.78	1.0847	20.75	22.50	1.0912	22.23	24.26	1.0977	23.63	25.94
1.0783	19.29	20.80	1.0848	20.77	22.53	1.0913	22.26	24.29	1.0978	23.65	25.97
1.0784	19.31	20.82	1.0849	20.79	22.55	1.0914	22.28	24.31	1.0979	23.67	25.99
1.0785	19.33	20.85	1.0850	20.81	22.58	1.0915	22.30	24.34	1.0980	23.69	26.01
1.0786	19.36	20.88	1.0851	20.83	22.61	1.0916	22.32	24.37	1.0981	23.71	26.04
1.0787	19.38	20.90	1.0852	20.86	22.64	1.0917	22.34	24.39	1.0982	23.73	26.06
1.0788	19.40	20.93	1.0853	20.88	22.66	1.0918	22.37	24.42	1.0983	23.76	26.09
1.0789	19.42	20.95	1.0854	20.90	22.68	1.0919	22.39	24.44	1.0984	23.78	26.11
1.0790	19.44	20.98	1.0855	20.93	22.72	1.0920	22.41	24.47	1.0985	23.80	26.14
1.0791	19.46	21.00	1.0856	20.95	22.75	1.0921	22.43	24.49	1.0986	23.82	26.17
1.0792	19.49	21.03	1.0857	20.98	22.78	1.0922	22.45	24.51	1.0987	23.84	26.19
1.0793	19.51	21.06	1.0858	21.01	22.81	1.0923	22.48	24.54	1.0988	23.86	26.22
1.0794	19.53	21.08	1.0859	21.04	22.84	1.0924	22.50	24.56	1.0989	23.88	26.24
1.0795	19.56	21.11	1.0860	21.06	22.87	1.0925	22.52	24.60	1.0990	23.90	26.27
1.0796	19.58	21.14	1.0861	21.09	22.90	1.0926	22.54	24.62	1.0991	23.92	26.30
1.0797	19.60	21.16	1.0862	21.11	22.93	1.0927	22.56	24.64	1.0992	23.94	26.32
1.0798	19.63	21.20	1.0863	21.13	22.96	1.0928	22.59	24.67	1.0993	23.97	26.35
1.0799	19.65	21.22	1.0864	21.16	22.99	1.0929	22.61	24.70	1.0994	23.99	26.37
1.0800	19.67	21.24	1.0865	21.19	23.02	1.0930	22.63	24.73	1.0995	24.01	26.40
1.0801	19.70	21.28	1.0866	21.22	23.06	1.0931	22.65	24.76	1.0996	24.03	26.42
1.0802	19.72	21.30	1.0867	21.25	23.09	1.0932	22.67	24.78	1.0997	24.05	26.44
1.0803	19.74	21.33	1.0868	21.28	23.12	1.0933	22.69	24.81	1.0998	24.07	26.47
1.0804	19.77	21.36	1.0869	21.30	23.15	1.0934	22.71	24.83	1.0999	24.09	26.49
1.0805	19.79	21.38	1.0870	21.33	23.18	1.0935	22.73	24.86	1.1000	24.11	26.52
1.0806	19.81	21.41	1.0871	21.35	23.21	1.0936	22.75	24.89	1.1001	24.13	26.55
1.0807	19.84	21.43	1.0872	21.37	23.23	1.0937	22.77	24.91	1.1002	24.15	26.57
1.0808	19.86	21.46	1.0873	21.39	23.26	1.0938	22.80	24.93	1.1003	24.17	26.60
1.0809	19.88	21.49	1.0874	21.41	23.28	1.0939	22.82	24.96	1.1004	24.19	26.62
1.0810	19.91	21.52	1.0875	21.43	23.31	1.0940	22.84	24.99	1.1005	24.21	26.65
1.0811	19.93	21.55	1.0876	21.45	23.33	1.0941	22.86	25.01	1.1006	24.23	26.68
1.0812	19.96	21.58	1.0877	21.47	23.36	1.0942	22.88	25.03	1.1007	24.25	26.70
1.0813	19.98	21.60	1.0878	21.49	23.38	1.0943	22.90	25.06	1.1008	24.28	26.73
1.0814	20.00	21.63	1.0879	21.51	23.40	1.0944	22.92	25.08	1.1009	24.30	26.75
1.0815	20.03	21.66	1.0880	21.54	23.43	1.0945	22.94	25.11	1.1010	24.32	26.78
1.0816	20.05	21.69	1.0881	21.56	23.45	1.0946	22.96	25.14	1.1011	24.34	26.81
1.0817	20.07	21.71	1.0882	21.58	23.48	1.0947	22.98	25.16	1.1012	24.36	26.83
1.0818	20.10	21.74	1.0883	21.60	23.50	1.0948	23.00	25.18	1.1013	24.39	26.86
1.0819	20.12	21.77	1.0884	21.62	23.52	1.0949	23.03	25.21	1.1014	24.41	26.88
1.0820	20.14	21.79	1.0885	21.64	23.55	1.0950	23.05	25.24	1.1015	24.43	26.91
1.0821	20.17	21.82	1.0886	21.66	23.58	1.0951	23.07	25.26	1.1016	24.45	26.93
1.0822	20.19	21.85	1.0887	21.68	23.60	1.0952	23.10	25.29	1.1017	24.47	26.95
1.0823	20.21	21.87	1.0888	21.71	23.63	1.0953	23.12	25.31	1.1018	24.49	26.98
1.0824	20.24	21.91	1.0889	21.73	23.66	1.0954	23.14	25.34	1.1019	24.51	27.00
1.0825	20.26	21.93	1.0890	21.75	23.69	1.0955	23.16	25.37	1.1020	24.53	27.03
1.0826	20.28	21.96	1.0891	21.77	23.72	1.0956	23.18	25.39	1.1021	24.55	27.06
1.0827	20.31	21.99	1.0892	21.79	23.74	1.0957	23.20	25.42	1.1022	24.57	27.08
1.0828	20.33	22.01	1.0893	21.82	23.77	1.0958	23.23	25.45	1.1023	24.60	27.11
1.0829	20.35	22.04	1.0894	21.84	23.79	1.0959	23.25	25.47	1.1024	24.62	27.14
1.0830	20.37	22.06	1.0895	21.86	23.82	1.0960	23.27	25.50	1.1025	24.64	27.17
1.0831	20.39	22.09	1.0896	21.89	23.85	1.0961	23.29	25.53	1.1026	24.66	27.19
1.0832	20.41	22.11	1.0897	21.91	23.87	1.0962	23.31	25.55	1.1027	24.68	27.21
1.0833	20.43	22.13	1.0898	21.93	23.90	1.0963	23.33	25.58	1.1028	24.71	27.24
1.0834	20.46	22.16	1.0899	21.96	23.93	1.0964	23.35	25.60	1.1029	24.72	27.26
1.0835	20.48	22.19	1.0900	21.98	23.96	1.0965	23.37	25.63	1.1030	24.74	27.29
1.0836	20.50	22.21	1.0901	22.00	23.98	1.0966	23.39	25.66	1.1031	24.76	27.32
1.0837	20.52	22.24	1.0902	22.02	24.01	1.0967	23.41	25.68	1.1032	24.78	27.34
1.0838	20.54	22.26	1.0903	22.04	24.03	1.0968	23.44	25.71	1.1033	24.81	27.37
1.0839	20.56	22.29	1.0904	22.06	24.05	1.0969	23.46	25.73	1.1034	24.83	27.39
1.0840	20.59	22.32	1.0905	22.08	24.08	1.0970	23.48	25.76	1.1035	24.85	27.42
1.0841	20.62	22.35	1.0906	22.10	24.11	1.0971	23.50	25.79	1.1036	24.87	27.45
1.0842	20.64	22.38	1.0907	22.12	24.13	1.0972	23.52	25.81	1.1037	24.89	27.47
1.0843	20.66	22.40	1.0908	22.15	24.16	1.0973	23.55	25.84	1.1038	24.92	27.50
1.0844	20.68	22.42	1.0909	22.17	24.18	1.0974	23.57	25.86	1.1039	24.94	27.53

EXTRACT IN BEER WORT—(*Concluded*).

Specific Gravity at 15° C.	Extract. Per Cent by Weight	Extract. Grams per 100 cc.	Specific Gravity at 15° C.	Extract. Per Cent by Weight	Extract. Grams per 100 cc.	Specific Gravity at 15° C.	Extract. Per Cent by Weight	Extract. Grams per 100 cc.	Specific Gravity at 15° C.	Extract. Per Cent by Weight	Extract. Grams per 100 cc.
1.1040	24.96	27.56	1.1095	26.16	29.03	1.1150	27.29	30.43	1.1205	28.38	31.81
1.1041	24.98	27.58	1.1096	26.18	29.06	1.1151	27.31	30.45	1.1206	28.40	31.83
1.1042	25.00	27.60	1.1097	26.20	29.08	1.1152	27.33	30.47	1.1207	28.42	31.86
1.1043	25.03	27.63	1.1098	26.23	29.11	1.1153	27.35	30.50	1.1208	28.44	31.88
1.1044	25.05	27.66	1.1099	26.25	29.13	1.1154	27.37	30.52	1.1209	28.46	31.90
1.1045	25.07	27.69	1.1100	26.27	29.16	1.1155	27.38	30.55	1.1210	28.48	31.93
1.1046	25.09	27.72	1.1101	26.29	29.19	1.1156	27.40	30.57	1.1211	28.50	31.95
1.1047	25.11	27.74	1.1102	26.31	29.21	1.1157	27.42	30.59	1.1212	28.52	31.98
1.1048	25.14	27.77	1.1103	26.33	29.24	1.1158	27.44	30.62	1.1213	28.54	32.00
1.1049	25.16	27.79	1.1104	26.35	29.26	1.1159	27.46	30.64	1.1214	28.56	32.03
1.1050	25.18	27.82	1.1105	26.37	29.29	1.1160	27.48	30.67	1.1215	28.58	32.05
1.1051	25.20	27.85	1.1106	26.39	29.32	1.1161	27.50	30.69	1.1216	28.60	32.08
1.1052	25.22	27.87	1.1107	26.41	29.34	1.1162	27.52	30.72	1.1217	28.62	32.11
1.1053	25.24	27.90	1.1108	26.44	29.37	1.1163	27.54	30.75	1.1218	28.64	32.13
1.1054	25.27	27.93	1.1109	26.46	29.39	1.1164	27.56	30.77	1.1219	28.66	32.15
1.1055	25.29	27.96	1.1110	26.48	29.42	1.1165	27.58	30.80	1.1220	28.68	32.18
1.1056	25.31	27.98	1.1111	26.50	29.44	1.1166	27.60	30.82	1.1221	28.70	32.20
1.1057	25.33	28.00	1.1112	26.52	29.46	1.1167	27.62	30.85	1.1222	28.72	32.23
1.1058	25.35	28.03	1.1113	26.54	29.49	1.1168	27.64	30.87	1.1223	28.74	32.25
1.1059	25.38	28.06	1.1114	26.56	29.51	1.1169	27.66	30.89	1.1224	28.76	32.27
1.1060	25.40	28.09	1.1115	26.58	29.54	1.1170	27.68	30.92	1.1225	28.78	32.30
1.1061	25.42	28.12	1.1116	26.60	29.57	1.1171	27.70	30.94	1.1226	28.80	32.32
1.1062	25.44	28.14	1.1117	26.62	29.59	1.1172	27.72	30.97	1.1227	28.82	32.35
1.1063	25.46	28.17	1.1118	26.64	29.61	1.1173	27.74	31.00	1.1228	28.84	32.37
1.1064	25.48	28.19	1.1119	26.66	29.64	1.1174	27.76	31.02	1.1229	28.86	32.40
1.1065	25.50	28.22	1.1120	26.68	29.67	1.1175	27.78	31.05	1.1230	28.88	32.43
1.1066	25.52	28.25	1.1121	26.70	29.69	1.1176	27.80	31.07	1.1231	28.90	32.45
1.1067	25.54	28.27	1.1122	26.72	29.71	1.1177	27.82	31.09	1.1232	28.92	32.48
1.1068	25.57	28.30	1.1123	26.75	29.74	1.1178	27.84	31.12	1.1233	28.94	32.50
1.1069	25.59	28.32	1.1124	26.77	29.77	1.1179	27.86	31.15	1.1234	28.96	32.53
1.1070	25.61	28.35	1.1125	26.79	29.80	1.1180	27.88	31.18	1.1235	28.98	32.56
1.1071	25.63	28.38	1.1126	26.81	29.83	1.1181	27.90	31.20	1.1236	29.00	32.58
1.1072	25.65	28.40	1.1127	26.83	29.85	1.1182	27.92	31.23	1.1237	29.02	32.60
1.1073	25.67	28.43	1.1128	26.85	29.88	1.1183	27.94	31.25	1.1238	29.04	32.63
1.1074	25.69	28.45	1.1129	26.87	29.90	1.1184	27.96	31.27	1.1239	29.06	32.65
1.1075	25.71	28.48	1.1130	26.89	29.93	1.1185	27.98	31.30	1.1240	29.08	32.68
1.1076	25.73	28.51	1.1131	26.91	29.95	1.1186	28.00	31.32	1.1241	29.10	32.71
1.1077	25.75	28.53	1.1132	26.93	29.97	1.1187	28.02	31.35	1.1242	29.12	32.73
1.1078	25.78	28.56	1.1133	26.95	30.00	1.1188	28.04	31.37	1.1243	29.14	32.76
1.1079	25.80	28.58	1.1134	26.97	30.02	1.1189	28.07	31.40	1.1244	29.16	32.78

are ascertained. This figure is added to the "extract gravity" to find the "original gravity of the wort."

SUGAR USED UP IN FERMENTATION.

Degrees of "Spirit Indication."	0.0000	0.0001	0.0002	0.0003	0.0004	0.0005	0.0006	0.0007	0.0008	0.0009
0.000	.....	0.0003	0.0006	0.0009	0.0012	0.0015	0.0018	0.0021	0.0024	0.0027
.001	.0030	.0033	.0037	.0041	.0044	.0048	.0051	.0055	.0059	.0062
.002	.0066	.0070	.0074	.0078	.0082	.0086	.0090	.0094	.0098	.0102
.003	.0107	.0111	.0115	.0120	.0124	.0129	.0133	.0138	.0142	.0147
.004	.0151	.0155	.0160	.0164	.0168	.0173	.0177	.0182	.0186	.0191
.005	.0195	.0199	.0204	.0209	.0213	.0218	.0222	.0227	.0231	.0236
.006	.0241	.0245	.0250	.0255	.0260	.0264	.0269	.0274	.0278	.0283
.007	.0288	.0292	.0297	.0302	.0307	.0312	.0317	.0322	.0327	.0332
.008	.0337	.0343	.0348	.0354	.0359	.0365	.0370	.0375	.0380	.0386
.009	.0391	.0397	.0402	.0407	.0412	.0417	.0422	.0427	.0432	.0437
.010	.0442	.0447	.0451	.0456	.0460	.0465	.0476	.0475	.0480	.0485
.011	.0490	.0496	.0501	.0506	.0512	.0517	.0522	.0527	.0533	.0538
.012	.0543	.0549	.0554	.0559	.0564	.0569	.0574	.0579	.0584	.0589
.013	.0594	.0600	.0605	.0611	.0616	.0622	.0627	.0633	.0638	.0643
.014	.0648	.0654	.0659	.0665	.0471	.0676	.0682	.0687	.0693	.0699
.015	.0705	.0711	.0717	.0723	.0729	.0735	.0741	.0747	.0753	.0759

Example.—Suppose the "extract gravity" is 1.0389 and the specific gravity of the alcoholic distillate is 0.9902, both at 15.6. Then $1-0.9902=0.0098$, the "degree of spirit indication." From the above table the corresponding "degree of gravity lost" is found to be 0.0432.

$0.0432+1.0389=1.0821$, the original gravity of the wort.

The calculation in the above simplified form is accurate for normal beer wherein the free acid present, expressed as acetic, does not exceed 0.1%. In case of beer that has developed free acid much in excess of the above limit, a correction should be added to the degrees of spirit indication. This correction, which in practice it is rarely necessary to apply except in extreme cases of old or sour beer, is calculated as follows:

If a represents the grams of free acid (as acetic) in 100 cc., then the correction to be added to the spirit indication $=0.0013a-0.00014$.

Example.—Supposing the "extract gravity" to be 1.0413, the specific gravity of the alcoholic distillate to be 0.9890, and the free acid as acetic to be 0.35%. Then $1-0.989=0.0110$, the degree of spirit indication.

$0.35\times0.0013-0.00014=0.0003$, correction to be added to the spirit indication.

$0.0110+0.0003=0.0113$, corrected spirit indication.

From the above table the corresponding degrees of gravity lost are 0.0506:

0.0506 + 1.0413 = 1.0919, the original gravity of the wort.

Determination of Total, Fixed, and Volatile Acids. — A measured volume of the beer, say 10 cc., is freed from carbon dioxide by bringing to boiling. It is then cooled and titrated with tenth-normal sodium hydroxide, using neutral litmus solution as an indicator. Each cubic centimeter of tenth-normal alkali is equivalent to 0.009 gram of lactic acid, in which the total acidity is usually expressed.

Fixed acid, also expressed as lactic, though small quantities of succinic, tannic, and malic acids are usually also present, is determined as follows: Dealcoholize a measured amount of the beer, say 10 cc., by evaporation to one-fourth its volume, dilute with water to the original volume, and titrate with tenth-normal alkali, as before.

Volatile acid is expressed as acetic, and is usually calculated by difference between total and fixed acid. Each cubic centimeter of tenth-normal alkali is the equivalent of 0.006 gram acetic acid.

Determination of Proteids.—Fifty cc. of the beer are first treated with 5 cc. of dilute sulphuric acid, and concentrated by boiling to syrupy consistency. Then proceed by the Gunning method, p. 61. $N \times 6.25 =$ proteids.

Determination of Phosphoric Acid.—Unless the sample is very dark-colored, sufficiently close results can usually be obtained by direct titration of the beer itself with uranium acetate solution. For very accurate results the ash should be used. Prepare a solution of uranium acetate of such strength that 20 cc. will correspond to 0.1 gram P_2O_5. This solution is best standardized against pure, crystallized, unefflorescd, powdered hydrogen sodium phosphate, 10.085 grams of which are dissolved in

drops of the solution to a porcelain plate, and touching with a drop of freshly prepared potassium ferrocyanide solution. When the slightest excess of uranium acetate has been added, a reddish-brown color is produced by the ferrocyanide. The uranium acetate solution is purposely made rather stronger than necessary at first, and by repeated trials is brought by dilution with water to the required strength (20 cc. equivalent to 50 cc. of the phosphate solution).

Fifty cc. of the beer are heated to 90° or 100° C. and titrated with the uranium acetate solution under the same conditions and in precisely the same manner as when standardizing that solution. Each cubic centimeter of the uranium acetate corresponds to 0.01% of P_2O_5.

For the phosphoric acid determination in the ash, 50 cc. of the beer are incinerated in the regular manner, and the ash moistened with concentrated hydrochloric acid. The acid is then evaporated off on the water-bath, after which the ash is boiled with 50 cc. of distilled water, and titrated with the standard uranium solution.

Determination of Carbon Dioxide.*—In the case of beer and other carbonated drinks put up in corked bottles, the carbon dioxide may be readily determined by piercing the cork with a metal champagne tap, which is connected by a flexible tube, first with a safety flask and then with an absorption apparatus somewhat after the style of that used in the determination of carbon dioxide in baking powder, the liberated carbon dioxide being absorbed for weighing in a concentrated solution of potassium hydroxide contained in a tared Liebig bulb. The beer-bottle thus connected is immersed in a vessel of water, which is heated over a gas-flame, after all the carbon dioxide that will escape spontaneously has been allowed to do so. Before weighing the absorbed carbon dioxide, the beer-bottle is replaced by a soda-lime tube, and a current of air drawn through the tubes.

Beer and ale put up in bottles having patent metallic or rubber stoppers cannot thus be treated. In this case a measured quantity, say 200 cc., of the sample is transferred as quickly as possible to a large flask provided with an outlet-tube having a glass stopper, this being connected up with the safety-flask and absorption-tubes. In this case heat may be directly, though cautiously, applied to the flask containing the beer by means of a gas-flame, after all the carbon dioxide has gone over that will do so spontaneously. Exactly the same apparatus as that shown in Fig.

* U. S. Dept. of Agric., Bur. of Chem., Bul. 65, p. 95.

65 may be used to advantage for determination of carbon dioxide in beer, except that a larger distilling-flask should be used in the case of beer.

Detection of Bitter Principles.—Elaborate schemes have been worked out for the systematic treatment of beer and ale for bitter principles. Nearly all of these are complicated and somewhat unsatisfactory. The presence of alkaloids in malt liquors, deliberately introduced during the process of manufacture, is now so rare that the analyst need seldom look for them, except in cases of suspected poisoning, when the scheme of Dragendorf* or of Otto-Stas should be employed. While it is somewhat difficult to positively identify the various alkaloids, it is usually easy to prove their absence in clear solutions, if on treatment with either of the general alkaloidal reagents, Mayer's solution (Reagent No. 170), or iodine in potassium iodide (Reagent No. 143), no precipitate is formed.

It is comparatively easy to prove the mere presence or absence of hop substitutes. The bitter principle of hops is readily soluble in ether, when a sample of the beer evaporated to syrupy consistency is extracted therewith, while the bitters of quassia and aloes, common hop substitutes, are insoluble in ether. Though many varieties of bitters might be employed that are soluble in ether, the *absence* of a bitter taste from the ether extract is evidence of the absence of hops.

The most marked difference analytically between hops and their substitutes in malt liquors lies in the fact that the bitter principle of hops is completely precipitated therefrom by treatment of the beer with lead acetate (either basic or neutral), leaving no bitter taste in the filtrate after concentration, while if any of the hop substitutes are present, the concentrated filtrate from the lead acetate treatment will have a bitter taste. The excess of lead should be removed from the filtrate, before concentration and tasting, by treatment with hydrogen sulphide. If the



On treatment of quassiin with bromine and sodium hydroxide or ammonia, a bright-yellow color is shown.

Chiretta is readily dissolved by ether from its aqueous solution. Its ether residue, when treated with bromine and ammonia, gives a straw color, slowly changing to a dull purple-brown. This is not true of its chloroform residue, so that it is not to be mistaken for quassia (Allen).

Gentian Bitter may be extracted by treatment of the acid liquor with chloroform. When the residue containing gentian bitter is treated with concentrated sulphuric acid in the cold, no color is produced, but on warming gently a carmine-red color is shown; if further treated with ferric chloride solution, a green-brown color is formed.

Aloes.—This substance is separated from beer by treating the dried residue from 200 cc. of the beer with warm ammonia, filtering, cooling, and treating the filtrate with hydrochloric acid. The resin of aloes is precipitated and collected on a filter. It is insoluble in cold water, ether, chloroform, or petroleum ether, but is soluble in alcohol. It has a very characteristic odor, which serves to identify it. The hot-water solution gives a curdy precipitate on treatment with lead acetate.

Capsicin is extracted by treatment of the acid liquor with chloroform. It is recognizable by its sharp, pungent taste.

Detection of Arsenic.—*By the Marsh Method.*—Measure 100 cc. of the beer (freed from carbon dioxide by agitation) into a seven-inch porcelain evaporating-dish, add 20 cc. pure concentrated nitric acid, and 3 cc. pure concentrated sulphuric acid, and cautiously heat till vigorous chemical action sets in, accompanied by frothing and swelling of the beer. Turn the flame low or remove it altogether, and stir vigorously till the frothing ceases, after which the liquid may be boiled freely. At this stage transfer to a large casserole, and continue the boiling till nearly all the nitric acid is driven off. Then, holding the casserole by the handle, continue the heating till the mass chars and the fumes of sulphuric acid are given off, giving the casserole a rotary motion to prevent sputtering. The residue should be reduced to a dry, black, pulverulent char soon after the sulphuric acid fumes begin to come off freely. If still liquid, pieces of filter-paper should be stirred in while still heating, till the residue is dry, avoiding an excess of paper.

Cool, add 50 cc. of water, and remove the masses of char from the sides of the dish by the stirring-rod. Heat to boiling and filter. Use the filtrate for the Marsh apparatus, adding it gradually.

The arsenic mirror may be weighed in the usual manner, if of sufficient size.

Reinsch's Test.*—Two hundred cc. of the beer are acidified with 1 cc. of pure, concentrated, arsenic-free hydrochloric acid, and evaporated to half its volume. 15 cc. more of hydrochloric acid are then added, and a piece of pure burnished copper foil half an inch long and a quarter of an inch wide is immersed in the liquid and kept in it for an hour while simmering, replacing from time to time the water lost by evaporation. If after the lapse of an hour the copper still remains bright, no arsenic is present.

If the copper shows a deposit, remove, wash with water, alcohol, and ether, and dry. Then place the copper in a subliming-tube, and heat over a low flame. Tetrahedral crystals, apparent under the microscope, show the presence of arsenic. Blackening of the copper does not in itself prove arsenic.

Malt Extract.—True malt extract is a syrupy fluid having a specific gravity of from 1.3 to 1.6, and made up in accordance with the following directions of the 1880 Pharmacopœia: Upon 100 parts of coarsely powdered malt contained in a suitable vessel, pour 100 parts of water, and macerate for six hours. Then add 400 parts of water, heated to about 30° C. and digest for an hour at a temperature not exceeding 55° C. Strain the mixture with strong pressure. Finally, by means of a water-bath or vacuum apparatus, at a temperature not exceeding 55° C., evaporate the strained liquid rapidly to the consistence of thick honey.

Keep the product in well-closed vessels in a cool place.

Such an extract has a residue of at least 70%, consisting chiefly of maltose, and contains about 2% of diastase. It should furthermore be capable of converting its own weight of starch at 55° C. in less than ten



There are on the market many so-called malt extracts widely advertised for their tonic and medicinal virtues, having the taste and consistency of beer or ale. In fact they are virtually beer, differing therefrom mainly in respect to price. Such "malt extracts" have no diastase, and their value as nutrients depends almost entirely on their sugar content.

Harrington * has analyzed twenty-one of the best known of these alleged malt extracts, the maximum, minimum, and mean results of his analyses being as follows:

	Specific Gravity.	Alcohol.	Total Residue.	Ash.
Maximum	1.0555	7.13	13.63	0.53
Minimum	1.0149	0.74	5.13	0.20
Mean	.....	3.94	8.78	

None of them contained any diastase, and several were preserved with salicylic acid.

DISTILLED LIQUORS.

These beverages differ from those hitherto considered, by reason of their high alcoholic content and low extract or residue. Indeed, when first distilled they are entirely without residue, but from long storage in casks, they absorb certain extractives from the wood, that impart more or less flavor as well as color.

When any fermented alcoholic infusion is subjected to distillation under ordinary circumstances, a distillate results which consists of a mixture with water of a large number of alcohols, chief among which is ethyl alcohol. The high boiling alcohols—amyl, butyl, propyl, etc., with their esters—exist in the distillate in small amount, constituting what is known as fusel oil. The various distilled liquors of commerce are made by just such a process of distillation, the product varying widely in flavor and character with the basis from which it was distilled.

By the processes involving the use of the so-called pot-still (the old-fashioned copper still and worm), it was impossible to obtain a pure concentrated ethyl alcohol from fermented grain mash, the basis of whiskey, except by many repeated distillations.

Now, however, by the use of improved apparatus, such as the Coffey still, involving the principle of fractional condensation, it is possible to obtain what is known as "silent spirit," or ethyl alcohol, free from

* Boston Medical and Surgical Journal, Dec. 31, 1896.

fusel oil. With proper appurtenances for rectifying, one can now obtain 95% alcohol by two distillations.

Composition of Fusel Oil.—Fusel oil varies considerably in composition with the source from which it is derived. Amyl alcohol, being in all cases its chief constituent, is frequently known commercially as fusel oil. The alcohols found in fusel oil with their formulas, specific gravity, and boiling-points are as follows:

	Formula.	Specific Gravity.	Boiling-point.
Ethyl alcohol	C_2H_5OH	.794	78.4° C.
Propyl "	C_3H_7OH	.820	97° C.
Butyl "	C_4H_9OH	.803	115° C.
Amyl "	$C_5H_{11}OH$	.811	130° C.
Hexyl "	$C_6H_{13}OH$		

The following acids have been found in fusel oil, usually combined with the alcohols to form compound ethers:

Acetic	$HC_2H_3O_2$	Caproic	$HC_6H_{11}O_2$
Propionic	$HC_3H_5O_2$	Œnanthylic	$HC_7H_{13}O_2$
Butyric	$HC_4H_7O_2$	Caprylic	$HC_8H_{15}O_2$
Valerianic	$HC_5H_9O_2$	Pelargonic	$HC_9H_{17}O_2$

Aging.—Freshly distilled liquors all contain notable quantities of fusel oil, which renders them harsh and unfit for use, but by the process of aging, the fusel oil becomes gradually transformed into the compound ethers that impart to the various liquors their distinctive bouquet, or flavor. Two or three years are usually required for the natural aging process.



Rarely malt alone is used, but more often the grain most abundant in the locality where the whiskey is distilled forms the basis of the liquor. Bourbon whiskey (made originally in Bourbon County, Kentucky) is made from a mixture of grain, 50 to 60 per cent of which is corn, 10% malt, and the balance rye.

Corn alone mixed with malt is employed in some localities, and pure rye whiskey is made from rye and malt. Scotch and Irish whiskey is commonly made from a mixture of oats, barley, rye, and malt.

Potatoes sometimes form the basis of the mash, the starch being converted to sugar either by mixture of the pulped potato with malt, or by boiling with dilute sulphuric acid and afterwards neutralizing, as in the manufacture of commercial glucose.

The fermented wort obtained from either source is subjected to distillation, without any attempt to rectify or separate out the fusel oil. The product of the first distillation is called "low wines" and is redistilled; the product of the second distillation is commonly divided into three fractions, of which the middle portion, or "clean spirit," is retained for the whiskey, while the first ("foreshots") and the last fraction (faints) are mixed with the next portion of low wine to be redistilled. If the whiskey is too high in alcohol, it is diluted to the proper strength. While the last fraction on redistillation is highest in fusel oil, notable quantities of the latter are present in the new whiskey, giving it a crude, harsh taste. It is therefore subjected to "aging," or storing in casks, preferably for a number of years. This aging process softens and refines the flavor, due to the transformation of the fusel oil into compound ethers. When first distilled whiskey is perfectly colorless, but during the aging it extracts more or less color and some flavor from the oak casks in which it is stored. Its flavor varies considerably with the nature of the grain used in its preparation. Malt dried over burning turf or peat (as used in the manufacture of Scotch whiskey) acquires a not unpleasant smoky taste, which it imparts to the finished product.

Composition.—Whiskey consists chiefly of alcohol and water, with the flavor due to its fusel oil derivatives. Its extract, derived mainly from the wine-cask in which it is stored, should consist only of traces of tannin, sugar, and coloring matter. It has a very small amount of volatile oil, rarely exceeding 0.1%.

Thirty-seven samples of whiskey, purchased by the glass from various Massachusetts saloons, were examined by the Massachusetts State Board of Health in 1894, with the following results:

	Per Cent Alcohol by Weight.	Per Cent Extract.
Maximum	45.96	1.68
Minimum	30.70	0.08
Mean	36.51	0.50

Seven of these samples had an excess of tannic acid, three had no tannic acid at all, two or three had insoluble residues, and one showed notable traces of fusel oil.

Adulteration of Whiskey and Standards of Purity.—The U. S. Pharmacopœia fixes the following requirements for whiskey: It should be at least two years old; in specific gravity it should lie between the limits of 0.930 and 0.917; its alcoholic content should be not less than 44% nor more than 50% by weight; its residue should be not more than 0.25%; the residue from 100 cc., which should be neither sweet nor spicy, should dissolve in 10 cc. of cold water, and this solution should be colored only a pale green when treated with a drop of very dilute ferric chloride solution (a deeper color would indicate more than traces of tannin). In evaporating the liquor on the water-bath for the residue, the last traces volatilized should have an agreeable odor free from harshness, indicative of the absence of fusel oil. Its reaction should be slightly acid, but not more than 1.2 cc. of normal alkali should be required to neutralize 100 cc. of the liquor, using phenolphthalein as an indicator.

The standards suggested for adoption to the A. O. A. C. by its Committee on Standards are as follows: Alcohol not less than 44% by volume nor more than 55%; solid matter not more than 0.25%.

Artificial Whiskey.—Whiskey is often artificially concocted by dilut-

"Beading oil" is prepared by mixing 48 ounces oil of sweet almonds with 12 ounces C. P. sulphuric acid, neutralizing with ammonia, adding double the volume of proof spirits, and distilling. This preparation is so called because it is largely used for putting an artificial bead on cheap liquors.

A little creosote is sometimes added to give a burnt taste in semblance of Irish whiskey. Pungent materials such as cayenne pepper are said to be used as adulterants, but no record is known of any substance being used more injurious than the alcohols. Sugar is a frequent adulterant.

Some doubt exists as to the injurious effects of fusel oil on the system.

BRANDY, OR COGNAC.

Brandy is the product of the distillation of fermented grape juice or wine. In a broader sense the term brandy is sometimes applied to liquor distilled from the juices of other fruits, such as apples, peaches, cherries, etc. The finest grades of brandy, such as pure cognac and armagnac (named from towns in France in which they were originally distilled), are made from choice white wine by the use of pot stills, and naturally command a high price. Brandy of a lower grade is distilled from the cheaper wines, and sometimes from the fermented marc, or refuse, of the grape, as well as from the lees and "scrapings" of the casks. The best brandies are sometimes rectified by a second distillation. Like whiskey, the fresh brandy is colorless, and would so remain if stored in glass or stone. The color is due to the wooden casks in which it is stored. Brandy consists of nearly pure alcohol and water, having a characteristic flavor, differing somewhat according to the nature and quality of the wine from which it was prepared. The chief flavor of pure cognac is due to œnanthic ether.

Thirty-seven samples of brandy, collected from Massachusetts barrooms in 1894 and examined by the State Board of Health, showed the following results:

	Per Cent Alcohol by Weight.	Per Cent Extract.
Maximum	50.70	3.00
Minimum	21.30	0.10
Mean	40.54	0.93

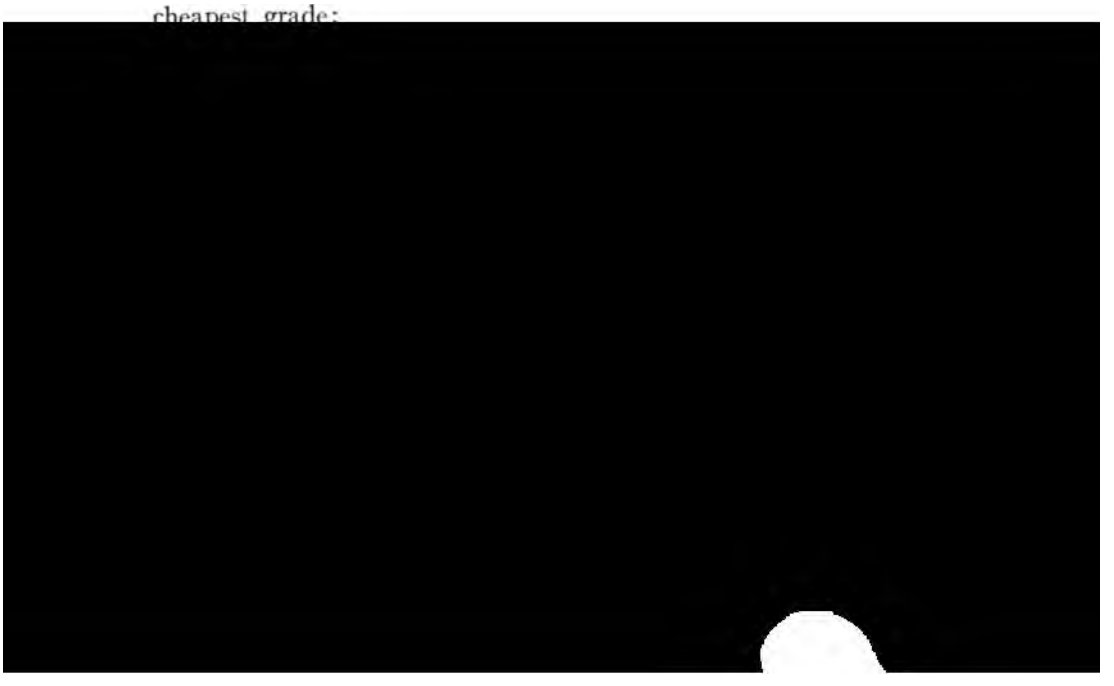
Three of these samples were artificially prepared mixtures of alcohol and water, one showed the presence of cloves, five contained tannin in excess, nine were excessively acid, two contained fusel oil, and two had insoluble residues.

Adulteration and Standards of Purity.—According to the U. S. Pharmacopœia, brandy should be at least four years old; its specific gravity should be not more than 0.941 nor less than 0.925; its alcoholic content should be from 39 to 47 per cent by weight; its residue should not exceed 1.5%; the residue from 100 cc. should dissolve readily in 10 cc. of cold water, and this solution should not be colored deeper than a pale green by the addition of dilute ferric chloride solution (absence of more than traces of tannin); the residue should not be sweet nor spicy in taste; a marked disagreeable pungent odor of fusel oil should not manifest itself on the volatilization of the last traces of alcohol in evaporating for the residue; in acidity, not more than 1 cc. of tenth-normal alkali should be required to neutralize 100 cc. of the brandy, using phenolphthalein as an indicator.

The suggested standards for the A. O. A. C. place the limits of alcohol between 44 and 55 per cent by volume, and the solid matter not to exceed 0.35%.

Much of the brandy sold on the market is of the "improved," or "blended," variety, having for its basis alcohol reduced to the requisite strength, flavored either by the admixture of real brandy, or by various preparations such, for example, as syrup of raisins, prune juice, rum, acetic ether, œnanthic ether, infusion of green walnut-hulls, infusion of bitter almond shells, catechu, balsam of Tolu, etc.

Fleischman gives the following recipe for artificial brandy of the cheapest grade:



Genuine new brandy may be "aged" or "improved" for immediate use, according to Duplais, by adding to 100 liters the following:

Old rum	2.00 liters
Old kirsch *	1.75 "
Infusion of walnut-hulls	.75 liter
Syrup of raisins	2.00 liters

The addition of sugar and caramel to brandy is very common. The lack of flavor resulting from the employment of "silent spirit," or from watering the product, may be compensated for by the employment of so-called cognac essences sold for the purpose, containing mixtures of the aromatic compounds named above.

RUM.

Rum is the liquor distilled from fermented molasses or cane juice, or from the scum and other waste juices from the manufacture of raw sugar. The molasses wort is mixed with the residue from a previous fermentation and allowed to ferment for a number of days, after which it is distilled twice and stored in wood for a long time, to remove the disagreeable odor, which in the new product is especially marked. The characteristic flavor of old rum is due to a mixture of butyric and acetic ether, principally the former. Pineapples and guavas are often put in the still to impart a fruity flavor. The best varieties of rum come from Jamaica and Vera Cruz.

Thirty-nine samples of rum, sold at retail in Massachusetts in 1894, were examined by the State Board of Health with the following results:

	Per Cent Alcohol by Weight.	Per Cent Extract.
Maximum	42.9	3.93
Minimum	24.7	0.04
Mean	37.1	0.51

Of these, two samples were new rum, and several were entirely artificial.

The suggested standard for rum of the A. O. A. C. sets the alcoholic content between 44 and 55 per cent by volume. More or less factitious rum is sold on the market, made up of alcohol diluted to the right strength,

* Brandy distilled from cherry wine.

colored with caramel, and flavored by the addition of "rum essence." Prune juice is sometimes added.

Fleischman gives the following recipe for low-grade artificial rum:

Spirits	40 gallons
New England rum	5 "
Prune juice	½ gallon
Caramel	12 ounces
Rum essence	8 "

The "rum essence" is made up by distilling 32 ounces of a mixture of 2 ounces black oxide of manganese, 4 ounces pyroligneous acid, 32 ounces alcohol, and 4 ounces sulphuric acid. To this is added 32 ounces of acetic ether, 8 ounces of butyric ether, 16 ounces saffron extract, and ⅛ ounce oil of birch.

GIN.

Gin is an alcoholic liquor, flavored with the volatile oil of juniper and sometimes with other aromatic substances, such as coriander, grains of paradise, anise, cardamom, orange-peel, and fennel. The choicest variety is known as Schiedam schnapps, named from the town of Schiedam in Holland, where there are upwards of 200 distilleries devoted to the manufacture of gin. The mash used for this variety is fermented by yeast from malted barley and rye, after which it is distilled and redistilled in pot stills with juniper berries and sometimes hops.

Juniper berries, to which the most characteristic flavor of gin is due, are dark blue in color, and possess a pungent taste. They grow on the slender evergreen shrub *Juniperus communis*. Gin differs from the other distilled liquors by being water-white. To this end it is kept in

METHODS OF ANALYSIS OF DISTILLED LIQUORS.—*Alcohol and Extract* are determined as directed on pages 528 and 547. Acidity is determined by titrating directly a measured volume of liquor, say 100 cc., with tenth-normal alkali, using phenolphthalein for an indicator and calculating results as acetic acid, 0.006 gram of which is the equivalent of 1 cc. of the standard alkali.

Detection of Fusel Oil.—In the process of dealcoholizing a liquor by evaporation in an open dish over the water-bath, one may readily detect fusel oil, if present, by its harsh and nauseating odor, if the nose is applied just at the moment when the last traces of alcohol are going off. At this stage any considerable trace of fusel oil will be especially apparent by the effect on the throat of the one who smells it, causing an uncontrollable desire to cough. Other ways of applying the odor test consist in pouring a small portion of the spirit into the hand, and allowing it to evaporate slowly therefrom, or in rinsing out a warm glass with the liquor, observing the odor in each case.

Goebel suggests the following test, based on the detection of the volatile acids: Agitate about 30 cc. of the liquor with 2 or 3 cc. of a dilute solution of potassium hydroxide; evaporate over the water-bath to the volume of 2 or 3 cc., cool, and to the residue add 5 or 6 cc. of concentrated sulphuric acid. If fusel oil be present, the characteristic odors of valerianic and butyric acids will be apparent.

Determination of Fusel Oil.—*The Röse-Stutzer-Windisch Method.**—The apparatus recommended for this determination is Bromwell's modification of Röse's fusel oil apparatus.

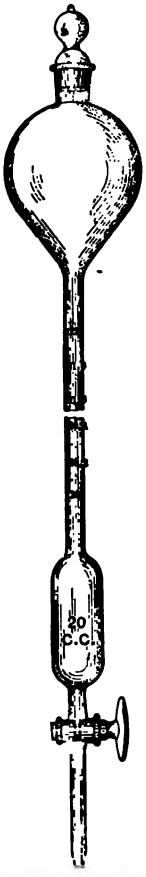
This apparatus consists of a pear-shaped bulb holding about 200 cc., stoppered at the upper end and sealed at the lower to a graduated stem about 4 mm. in internal diameter. To the lower end of this graduated stem is a sealed bulb of 20 cc. capacity, the lower end of which bears a stop-cock tube. The apparatus is graduated to 0.02 cc. from 20 cc. to 22.5 cc.

The reagents required are fusel-free alcohol, that has been prepared by fractional distillation over caustic soda or caustic potash and diluted to exactly 30% by volume (specific gravity 0.96541), chloroform freed from water and redistilled, and sulphuric acid (specific gravity 1.2857 at 15.6°).

Distill slowly 200 cc. of the sample under examination (having first

* Arbeit. d. kaiserl. Gesund. 1889, 5, 391. Zeits. f. anal. Chem., 34 (1895), Amtl. Verordn. u. Erl. S. 2. U. S. Dept. of Agric., Bur. of Chem., Bul. 46, p. 69.

made alkaline) till about 175 cc. have passed over, allow the distilling-flask to cool, add 25 cc. of water, and distill again till the total distillate measures 200 cc. Dilute the distillate to exactly 30% by volume (specific gravity 0.96541 at 15.6°).



Prepare a water-bath the contents of which are kept at exactly 15°, and place in it the apparatus (covering the end of the tube with a rubber cap to prevent wetting the inside of the tube) and flasks containing the 30% fusel-free alcohol, chloroform, sulphuric acid, and the distillate diluted to 30% by volume. When the solutions have all attained the temperature of 15°, fill the apparatus to the 20-cc. mark with the chloroform, drawing it through the lower tube by means of suction, add 100 cc. of the 30% fusel-free alcohol and 1 cc. of the sulphuric acid, invert the apparatus and shake vigorously for two or three minutes, interrupting once or twice to open the stop-cock for the purpose of equalizing pressure. Allow the apparatus to stand ten or fifteen minutes in water that is kept at the temperature of 15°, turning occasionally to hasten the separation of the reagents, and note the volume of the chloroform. After thoroughly cleansing and drying the apparatus, repeat this operation, using the diluted distillate from the sample under examination in place of the fusel-free alcohol. The increase in the chloroform volume with the sample under examination over that with the fusel-free alcohol is due to fusel oil, and this difference (expressed in cubic centi-



Determination of Ethereal Salts. — *Provisional Method of the A. O. A. C.*—Neutralize the residue left after distillation in the fusel oil determination with N/10 H_2SO_4, and add an excess of 10 cc. of the acid. Let stand 5 minutes and make up to 200 cc. Titrate 2 portions of 25 cc. each, using as indicators methyl orange in the first and phenolphthalein in the second. The difference gives the amount of alkali necessary to neutralize the organic acids in 25 cc. of the sample. By subtracting from this figure the number of cubic centimeters of alkali required for the free acids, and multiplying the result by 0.0088, the number of grams of ethereal salts (calculated as ethyl acetate) in 25 cc. of the sample is determined.

Furfurol in Distilled Liquors and its Determination.—Furfurol is found in liquors distilled from starchy materials in pot stills, or those heated by direct contact with the fire, but not in liquors produced by indirect or steam boiling. Hence the furfurol is believed to be due to the charring of these starchy ingredients.

McGill * describes the following colorimetric test: Fifty cc. of the liquor to be tested are measured into a Nessler tube, and 1 cc. of anilin reagent † is added. In the presence of furfurol a reddish color is produced, dependent in depth on the amount present, but readily observable with 0.001%. Results are reported as distinct, faint, or none, though approximate quantitative results are possible, if the color is matched against a known-strength solution of furfurol similarly treated.

Detection of Caramel.—*Crampton and Simons' Methods.*—(*a*) *With Fullers' Earth.*‡—Mix by vigorous shaking in a corked flask 50 cc. of the liquor with 25 grams of fullers' earth, let stand for half an hour, and filter. A naturally colored liquor will show little change in color in the filtrate, while a liquor colored with caramel will be largely decolorized, or at least deprived of such of the color as is due to caramel. By comparing in test-tubes the relative lengths of column of the filtrate, and the original liquor diluted with water to match the tint of the filtrate when viewed from above, one can approximately estimate the amount of caramel used.

All varieties of fullers' earth do not possess the property of absorbing

---

* Bul. 27 Canada Inland Rev. Dept.

† The reagent used is made up as follows:

Redistilled anilin	1 volume
Glacial acetic acid	1 "
Distilled water	2 volumes

‡ Jour. Am. Chem. Soc., 21 (1899), p. 355.

caramel in equal degree. When a satisfactory variety is obtained, it is well to procure a large amount for reagent purposes.

(*b*) ***With Ether.****—Evaporate 50 cc. of the liquor nearly but not quite to dryness on the water-bath. Wash with water into a 50-cc. graduated glass-stoppered flask, add 25 cc. of absolute alcohol, and fill to the mark with water. Shake, and transfer 25 cc. of the solution to a separatory funnel of the type represented in Fig. 116, the stem of which terminates in a 25-cc. graduated bulb pipette, provided with a stop-cock as shown.



FIG. 116.—Separatory Funnel for Detection of Caramel.

Add 50 cc. of ether, and shake carefully at intervals during half an hour. After complete separation, make up the lower aqueous layer with water to the 25-cc. mark, which may be done by siphoning it in through a rubber tube from an elevated flask, controlling the supply by the stop-cock. Shake the separatory funnel, and again allow the layers to separate, draw off the aqueous layer, and compare with the color of the original liquor. Ether will readily dissolve the natural color due to oak-wood (mainly flavescin), while caramel is insoluble in ether. Hence ether acts in a reverse manner to the fullers' earth, partially decolorizing naturally colored liquor, and showing little change of color with samples treated with caramel. Less variation in color between the original and final solution is shown by the ether treatment than by that with fullers' earth, though, according to Crampton and Simons, the two methods are always confirmatory.

Opalescence in Diluted Alcohol Distillate.—McGill † has shown that

bidity certainly furnishes a rough distinguishing test, indicating in some cases the exclusive use of rectified spirit.

LIQUEURS AND CORDIALS.

These are manufactured beverages, usually high in alcohol and sugar, flavored with a wide variety of aromatic herbs or essences, and often strongly colored. Red colors most frequently used for this purpose are cochineal, cudbear, and red sandal and Brazil woods; for yellow colors, caramel and saffron-yellow are employed; for blue, indigo; and for green, chlorophyll and malachite green.

Some of the oldest of the liqueurs, such as chartreuse and bénédictine, derive their names from certain monasteries of Europe, in which they have been made for many years.

Absinthe is one of the best-known cordials, made by redistilling 40% alcohol in which wormwood, anise, sweet flag, and marjoram leaves have been macerated. Sometimes coriander and fennel are also used. It is highly intoxicating.

Curaçoa is made by distilling dilute spirits in which Curaçoa orange-peel,* cinnamon and often other spices have been soaked, and by adding sugar to the resulting liqueur.

De Brevans gives the following recipe for curaçoa:

Rasped skins of.....	18 or 20 oranges
Cinnamon.....	4 grams
Mace.....	2 "
Alcohol (85%).....	5 liters
White sugar.....	1750 grams

Macerate for fourteen days, distill without rectification, and color with caramel.

Angostura owes its flavor to Angostura bark and various spices.

Maraschino had originally for its basis the fermented juice of the sour Italian cherry, to which honey was added. It is more commonly made by distilling a mixture in alcohol of ripe wild cherries, raspberries, cherry leaves, peach nuts, and orris. Finally sugar is added.

Chartreuse and Bénédictine contain much sugar, and are flavored with the volatile oils of angelica, hyssop, nutmeg, and peppermint.

Noyau, or Crême de Noyau, is a preparation distilled from brandy,

* This is a very rare and highly prized orange, growing in the island of Curaçoa.

bitter almonds, mace, and nutmeg. Sugar and coloring matter, usually pink, are added to the final product.

Crême de Menthe, according to De Brevans, is made by distilling a mixture of

Peppermint	600	grams
Balm	40	"
Sage	10	"
Cinnamon	20	"
Orris root	10	"
Ginger	15	"
Alcohol (80%)	5030	cc.

producing finally 10 liters of the liquor, after 3750 grams of white sugar have been introduced.

The green color of crême de menthe is almost entirely artificial. The better grades are colored with an alcoholic solution of chlorophyll, derived by macerating bruised green leaves of various plants with alcohol for several days. Coal-tar dyes are used in the cheaper grades.

The following analyses, due to König, show the chemical composition of the best-known cordials:

	Specific Gravity.	Alcohol by Volume.	Alcohol by Weight.	Extract.	Cane Sugar.	Other Extractives.	Ash.
Absinthe	0.9116	58.93	.....	0.18	.....	0.32	
Bénédictine	1.0709	52	38.5	36.00	32.57	3.43	0.043
Ginger	1.0481	47.5	36.0	27.79	25.92	1.87	0.141
Crême de menthe	1.0447	48.0	36.5	28.28	27.63	0.65	0.068
Anisette de Bordeaux	1.0847	42.0	30.7	34.82	37.44	0.38	0.040
Curaçoa	1.0300	55.0	42.5	28.60	28.50	0.10	0.040
Kümmel	1.0830	33.9	24.8	32.02	31.18	0.84	0.058

oils is shown, if on treatment of a few cubic centimeters of the sample in a test-tube with water a precipitate is formed.

GENERAL REFERENCES ON ALCOHOLIC BEVERAGES.

(See also References on Leavening Materials, page 275.)

BERSCH, J. Gahrungs-Chemie für Praktiker. Berlin. Vol. I, Die Hefe und die Gahrungs Erscheinungen, 1879. Vol. II, Fabrikation von Malz, Malz Extract und Dextrin, 1880. Vol. III, Die Bierbrauerei, 1881.

BIGELOW, W. D. Fermented and Distilled Liquors. U. S. Dept. of Agric., Bur. of Chem., Bul. 65, p. 81. 1902.

BOURGUELOT, E. Des Fermentations. Paris, 1889.

BREVANS, J. DE. The Manufacture of Liquors and Preserves. New York, 1893.

CRAMPTON, C. A. Fermented Alcoholic Beverages. U. S. Dept. of Agric., Div. of Chem., Bul. 13, part 3. 1887.

DUPLAIS, P. (Translated by McKennie, M.) A Treatise on the Manufacture and Distillation of Alcoholic Liquors. Philadelphia.

FLEISCHMAN, J. The Art of Blending and Compounding Liquors and Wines. New York, 1885.

GIRARD, C. La Fabrication des Liqueurs et des Conserves. Paris, 1890.

HANSEN, E. CH. Untersuchungen aus der Praxis der Gahrungs-Industrie. München, 1889.

MEW, J., and ASHTON, J. Drinks of the World. London, 1892.

PASTEUR, M. Studies in Fermentation. London, 1879.

PRESCOTT, A. B. Critical Examination of Alcoholic Liquors. New York, 1880.

SPENCER, E. The Flowing Bowl. A Treatise on Drinks of all Kinds and of all Periods. London, 1899.

STEVENSON, T. A Treatise on Alcohol with Tables of Spirit Gravities. London, 1888.

—— A Treatise on the Manufacture, Imitation, Adulteration and Reduction of Foreign Wines, Brandies, Rums and Gins, based upon the "French System," by a Practical Chemist and Experienced Liquor Dealer.

REFERENCES ON BEER.

ALLEN, A. H., and CHATTAWAY, W. Detection of Hop Substitutes in Beer. Analyst, 12, 1887, p. 107; also Analyst 15, 1890, p. 181.

BREVANS, J. DE. Analyse des Matières Alimentaires (Girard et Dupré), p. 183. Paris, 1894.

ELION, H. Detection of Antiseptics in Beer. Analyst, 16, 1891, p. 116.

FAULKNER, F. Theory and Practice of Modern Brewing. London, 1888.

HEFELMANN, R., and MANN, P. Detection of Fluorine in Beer. Pharm. Centralh., 1895, 16, 249; Abs. Analyst, 20, 185.

KELYNACK, T. N., and KIRBY, W. Arsenical Poisoning in Beer Drinkers. London, 1901.

LINDET, L. La Biere. Paris, 1892.

LINDNER, C. Lehrbuch der Bierbrauerei. Braunsweig, 1878.

MACFARLANE, T. Malt Liquors. Canada Inl. Rev. Dept. Bul. 52.

PASTEUR, M. Etudes sur la Biere. Paris, 1876.
PIESSE, C. H. Chemistry in the Brewing Room. London, 1891.
PRIOR, E. Chemie und Physiologie des Malzes und des Bieres. Leipzig, 1896.
STIERLEIN, R. Das Biere und seine Verfälschungen. Berlin, 1878.

REFERENCES ON CIDER AND WINE.

ALWOOD, W. B. A Study of Cider Making. U. S. Dept. of Agric., Bur. of Chem., Bul. 71.
BARILLOT, E. Manuel de l'Analyse des Vins. Paris, 1889.
BARTH, M. Die Weinanalyse. Leipzig, 1884.
BASTIDE, E. Les Vins Sophistiques. Paris, 1889.
BROWNE, C. A. The Chemical Analysis of the Apple, and some of Its Products. Jour. Am. Chem. Soc., 23, 1901, p. 869.
BORGMANN, E. Anleitung zur chemischen Analyse des Weines. Wiesbaden, 1898.
CAZENEUVE, P. La Coloration des Vins par les Couleurs de la Houille. Paris, 1886.
EMBREY, G. A Comparison of English and American Cider, with Suggestions for Estimating the Amount of Added Water. Analyst, 16, 1891, 41.
GAUTIER, A. La Sophistication des Vins. Paris, 1884.
MACFARLANE, T. Wines. Canada Inl. Rev. Dept. Bul. 38.
NESSLER, J. Die Bereitung, Pflege und Untersuchung des Weins. Stuttgart, 1889.
NIVIERE, G., and HUBERT, A. Detection of Fluorine in Wine. Monit. Scient., 1895, 9, p. 324; Abs. Analyst, 20, 185.
PASTEUR, M. Etudes sur le Vin. Paris, 1873.
ROBINET, E. Manuel Pratique d'Analyse des Vins. Paris, 1888.
SANGLE-FERRIERE. Vin. Analyse des Matières Alimentaires (Girard et Dupré), p. 65. Paris, 1894.
—— Cidre. Loc. cit., p. 217.
VIARD, E. Traité général des Vins et de leurs Falsifications. Paris, 1884.

REFERENCES ON DISTILLED LIQUORS.

ALLEN, A. H. The Chemistry of Whiskey and Allied Products. Jour. Soc. Chem. Ind., 10, 1891, p. 312.



CHAPTER XV.

VINEGAR.

VINEGAR is the product formed by the acetic fermentation of an alcoholic liquid under the influence of the organism *mycoderma aceti*, existing in the "mother-of-vinegar." While vinegar may be made directly from a dilute solution of pure alcohol, it is more often obtained from fruit juice, wine, or other saccharine liquid that has first undergone alcoholic fermentation.

Of the following equations, (1) and (2) illustrate the processes of inversion and alcoholic fermentation respectively, while (3) and (4) show the double process of acetic fermentation, wherein the alcohol is oxidized, first to acetaldehyde and finally to acetic acid:

$$\underset{\text{Cane sugar}}{C_{12}H_{22}O_{11}} + H_2O = 2\underset{\text{Invert sugar}}{C_6H_{12}O_6}; \quad \ldots\ldots (1)$$

$$\underset{\substack{\text{Invert sugar,}\\ \text{dextrose, or}\\ \text{maltose}}}{C_6H_{12}O_6} = 2\underset{\text{Alcohol}}{C_2H_6O} + 2CO_2; \quad \ldots\ldots (2)$$

$$\underset{\text{Alcohol}}{C_2H_6O} + O = \underset{\text{Aldehyde}}{C_2H_4O} + H_2O; \quad \ldots\ldots (3)$$

$$\underset{\text{Aldehyde}}{C_2H_4O} + O = \underset{\text{Acetic acid}}{C_2H_4O_2}. \quad \ldots\ldots (4)$$

In addition to the acetic acid, its chief active principle, vinegar usually contains traces of other organic acids free or combined, small amounts of alcohol, aldehyde, sugar, glycerin, coloring matter, aromatic ethers, and mineral salts, its extract varying considerably with the source from which the vinegar was obtained.

Varieties.—The principal varieties of vinegar are the following: *Cider vinegar*, *wine vinegar*, *malt or beer vinegar*, *spirit vinegar*, *glucose vinegar*, *molasses vinegar*, and *wood vinegar*, the three last being more frequently used as adulterants of the others.

Manufacture of Vinegar.—Cider vinegar, the principal variety used in the United States and Canada, was formerly made almost entirely by the slow process of cask fermentation, the fresh cider being allowed to undergo both alcoholic and acetic fermentation in barrels with open bung-holes in a warm cellar, or exposed to the sun. Two or three years are required for this process. Sometimes fresh cider is added to the barrels at regular intervals of two or three weeks, thus causing a series of progressive fermentations. The acetic fermentation is hastened by adding old vinegar, or mother-of-vinegar to the cider. While farmers and some manufacturers still continue to make cider vinegar by the slow process, the quick or "generator" vinegar process is now much used for cider vinegar, though originally intended and almost exclusively used in the manufacture of malt, beer, and spirit vinegar. This process requires only two or three days for complete acetification. In the quick process, the cider or other alcoholic liquor is allowed to percolate slowly through beech-wood shavings or birch twigs, held in a cask known as a generator, provided with a perforated, false bottom, the shavings or twigs being previously saturated with old vinegar, and a current of air being passed up through them.

The alcoholic liquid from which genuine malt vinegar is made is derived from the wort obtained by mashing malt, or a mixture of malt and barley. Spirit vinegar is derived from diluted whiskey, brandy, or grain alcohol. Wine vinegar is made by allowing the wine to stand over wine lees for a time, after which it is clarified by passing through beech shavings, and subjected to progressive acetification in large open oak casks, to which the wine is added, the vinegar being drawn off in much the same manner as the slow-process cider vinegar.

CHARACTERISTICS AND COMPOSITION OF THE VARIOUS VINEGARS.



	Acetic Acid.	Total Solids.	Ash.	Alkalinity of Ash.[1]	P_2O_5 in Ash of 100 Grams Vinegar.	
					Soluble (mgr.).	Insoluble (mgr.).
Maximum	5.86	3.20	0.42	36.1	31.7	31.5
Minimum	3.92	1.84	0.20	22.2	12.1	6.5
Average	4.84	2.49	0.34	29.7	19.2	15.6

	Reducing Sugars.		Polarization, Degrees Ventzke 200-mm. Tube.	Malic Acid.	Per Cent Ash in Total Solids.	Per Cent Reducing Sugars in Total Solids.	Ratio of Soluble to Total P_2O_5.	Alkalinity of 1 Gram of Ash, cc. $\frac{N}{10}$ Acid.
	Before Inversion.	After Inversion.						
Maximum	0.51	0.53	−3.6	0.16	19.0	16.6	66.9	125.0
Minimum	0.15	0.15	−0.3	0.08	10.0	7.3	50.0	69.0
Average	0.25	0.25	−1.3	0.11	13.8	10.7	56.3	90.0

[1] Number of cubic centimeters of tenth-normal acid to neutralize the ash of 100 grams of vinegar.

Twenty-two samples of pure cider vinegar were analyzed by A. W. Smith * with the following results:

	Acetic Acid.	Total Solids.	Ash.	Alkalinity of Ash.[1]	Soluble P_2O_5.	Insoluble P_2O_5.	Total P_2O_5.
Maximum	7.61	4.45	0.51	55.2	22.7	19.4	39.0
Minimum	3.24	2.00	0.31	28.4	13.6	4.2	19.8
Average	4.46	2.83	0.39	38.8	19.1	10.1	28.6

[1] Number of cubic centimeters of tenth-normal acid required to neutralize the ash from 100 grams of vinegar.

The composition of cider vinegar ash is found by Doolittle and Hess † to be as follows:

Calcium oxide	CaO	3.4 to 8.21
Magnesium oxide	MgO	1.88 " 3.44
Potassium oxide	K_2O	46.33 " 65.64
Sodium oxide	Na_2O	None
Sulphuric anhydride	SO_3	4.66 to 16.29
Phosphoric anhydride	P_2O_5	3.29 " 6.66
Iron oxide	Fe_2O_3	None " trace
	CO_2 and loss	0.00 " 40.44

Wine Vinegar is light yellow if made from white wine, and red if from red wine. The former is the highest prized. Wine vinegar varies in specific

* Jour. Am. Chem. Soc., 20 (1898), p. 6.

† *Ibid.*, 22 (1900), p. 220.

gravity from 1.0129 to 1.0213, and contains from 6 to 9 per cent of acetic acid. It is characterized chiefly by the bitartrate of potassium (cream of tartar) which true wine vinegar always possesses. Free tartaric acid is also usually present. Wine vinegar is the principal vinegar of France and Germany. In the United States the term white wine vinegar is usually applied to distilled or spirit vinegar, which is much cheaper than the real wine vinegar and altogether inferior to it.

Wine vinegar is slightly lævo-rotary with polarized light.

The composition of genuine white wine vinegar is shown by the following summary of the analyses of twenty-two samples, made in the Municipal Laboratory of Paris:

	Specific Gravity.	Total Solids.	Sugar.	Bitartrate of Potash.	Ash.	Acidity (as Acetic).
Maximum.....	1.0213	3.19	0.46	0.36	0.69	7.38
Minimum.....	1.0129	1.38	0.06	0.07	0.16	4.44
Mean.....	1.0175	1.93	0.22	0.17	0.32	7.38

Weigmann gives the following average of analyses of red wine vinegar:

Specific Gravity.	Acetic Acid.	Total Tartaric Acid.	Free Tartaric Acid.	Cream of Tartar.	Alcohol.	Extract.	Glycerin.	Ash.	Phosphoric Acid.
1.0143	7.79	0.216	0.006	0.057	1.19	0.863	0.141	0.118	0.012

Malt or Beer Vinegar is of a brown color, and its odor is suggestive of sour beer. It varies in specific gravity from 1.015 to 1.025; its acidity is about the same as cider vinegar, but the extract is much larger, varying

Allen gives the results of the analyses of three samples of genuine vinegar brewed from a mixture of malted and unmalted barley as follows:*

	Specific Gravity.	Acetic Acid.	Total Solids.	Ash.	Alkalinity as K_2O.	Phosphoric Acid.	Nitrogen.	Albuminoids.
1.....	1.0170	6.39	2.67	0.34	0.091	0.077	.099	.624
2.....	1.0228	5.26	3.96	0.40	0.118	0.093	.095	.598
3.....	1.0160	4.86	2.31	0.47	.....	0.057	.099	.624

Distilled, Spirit, or Alcohol Vinegar.—This vinegar, being made from diluted alcohol, is nearly colorless, unless artificially colored, as it often is, with caramel. As stated on page 612, the "white wine" vinegar (incorrectly so-called) commonly sold in the United States is of this class. Its specific gravity ranges from 1.008 to 1.013. Spirit vinegar contains from 3 to 10 per cent of acetic acid. Its content of total solids is insignificant, and it contains only traces of ash. It always contains non-acetified alcohol and aldehyde. It has no optical activity with polarized light.

Twelve samples of alcohol vinegar analyzed in the Municipal Laboratory of Paris gave the following results:

	Specific Gravity.	Total Solids.	Sugar.	Ash.	Acidity.
Maximum.....	1.0131	0.16	Trace	.09	7.98
Minimum.....	1.0082	0.07	"	.04	4.98
Mean.....	1.0100	0.35	"	Trace	6.34

Glucose Vinegar is made from the acetification of alcohol, obtained from the fermentation of commercial glucose. This vinegar usually possesses the odor and taste of fermented starch. It is low in total solids, the extract consisting almost entirely of untransformed glucose, and the vinegar therefrom contains all the ingredients of the product from which it was made, viz., dextrin, maltose, and dextrose, as well as sulphate of calcium. It is decidedly dextro-rotary with polarized light both before and after inversion.

Molasses Vinegar.—This is largely the product of the acetic fermentation of sugar-house wastes, and sometimes of the accidental acetic fermentation of molasses itself, after it has undergone alcoholic fermentation for the manufacture of rum. This variety of vinegar is sometimes

* Analyst, 19, p. 15.

used as an adulterant of cider vinegar. With polarized light molasses vinegar is dextro-rotary before, and lævo-rotary after inversion.

Wood Vinegar is prepared by the purification of pyroligneous acid, which may be accomplished by saturating the crude acid with lime or soda, adding hydrochloric or sulphuric acid, and distilling. It is further purified by redistillation with potassium bichromate, and filtration through bone-black. Acetic acid is sometimes added to impart flavor.

The extract and ash of wood vinegar are very small. Its specific gravity averages 1.007 according to Blyth. Empyreumatic or tarry products are nearly always present in vinegar of this class.

ANALYSIS OF VINEGAR.

Specific Gravity.—This is obtained either with the hydrometer, pycnometer, or Westphal balance.

Determination of Extract or Total Solids.—Weigh 5 grams of the sample in a tared platinum dish, and evaporate to dryness over the live steam of a boiling-water bath, keeping the dish thereon for two hours. Cool in a desiccator and weigh.

Determination of Ash.—Transfer the dish containing the last residue or extract to a muffle, and burn at a low red heat to an ash, or the ignition may be accomplished with care over a direct flame turned low. Cool the dish and weigh.

Determination of Solubility and Alkalinity of the Ash.—*Smith's Method.**—Twenty-five cc. of the vinegar are evaporated to dryness in a tared platinum dish, ignited, cooled, and the ash weighed. The ash is then repeatedly extracted with hot water by washing into a Gooch crucible provided with a layer of asbestos (previously ignited in the crucible, cooled, and weighed) or upon an ash-free filter. Dry the Gooch or filter, ignite

soluble ash. Precipitate the phosphoric acid from the two solutions separately by adding to each while hot an excess of ammonium molybdate (reagent No. 53). Digest for an hour at a temperature of about 65°, filter, and wash with a 10% solution of ammonium nitrate. Dissolve the precipitate on the filter with ammonia and hot water, and wash into a beaker to a bulk of not more than 100 cc. Nearly neutralize with hydrochloric acid, cool, and add slowly magnesia mixture (reagent No. 164) drop by drop while stirring vigorously. After fifteen minutes add 30 cc. of ammonia (specific gravity 0.96), let stand for two hours, filter, wash with 2.5% ammonia till practically free from chlorides, ignite, and weigh as $Mg_2P_2O_7$.

Express results in terms of milligrams of phosphoric anhydride in the soluble and insoluble vinegar ash from 100 grams of vinegar.

Phosphoric acid in the soluble and insoluble ash may be conveniently determined also by the uranium acetate method, page 588.

Determination of Nitrogen.—Concentrate from 50 to 100 cc. of vinegar to a syrupy consistency, and proceed as directed under the Gunning method, page 61.

Determination of Total Acidity.—Six cc. of vinegar are carefully measured from a pipette into a white porcelain dish and diluted with water. Using phenolphthalein as an indicator, titrate with tenth-normal sodium hydroxide. The number of cubic centimeters of the latter required to neutralize, divided by 10, expresses the acidity in terms of percentage of acetic acid.

Approximate Determination of Vinegar Acidity by Lime Water. — It has generally been considered difficult for vinegar dealers and others who desire to estimate the acidity of their vinegar to do this themselves, in that it has been necessary to obtain for the purpose a carefully standardized alkaline solution, the exact strength of which it is impossible for them to determine.

It has been found that very satisfactory, though of course not absolutely accurate, results may be obtained by the use of ordinary lime water, which any one may easily prepare by making a saturated solution of ordinary air-slaked lime. The strength of such a solution is very nearly constant, and has been found to be about $\frac{1}{40}$ of the normal. If, therefore, it is not easy to obtain exactly normal or tenth-normal alkali, approximate figures may be obtained by employing such a saturated lime water. If 2.75 cc. of vinegar are titrated with lime water contained in a burette, using phenolphthalein as an indicator, the number of cubic centimeters

of the lime water necessary to neutralize the vinegar, divided by 10, gives the percentage of acetic acid in the vinegar. To make sure that the lime water is saturated, an excess of lime should always be present in the reagent bottle.

Determination of Volatile and Fixed Acids.—Thirty cc. of the vinegar are transferred to a distilling-flask and subjected to distillation, using a current of steam. Receive the distillate in a 25-cc. graduated cylinder. After 15 cc. have passed over, test from time to time the drops of distillate as they fall into the receiving vessel with litmus-paper, and when free from acid discontinue the distillation. Note the volume of the distillate, mix by shaking, and transfer one-fifth to a white porcelain dish. Titrate as in the case of total acidity, expressing the volatile acids as acetic.

Calculate the fixed acid, expressed in the case of cider vinegar as malic, by subtracting the percentage of volatile acid from the percentage of total acid, and multiplying the result by the factor 1.117. In the case of wine vinegar, express as tartaric acid by using the factor 1.25. To express acidity in terms of sulphuric acid, multiply the percentage of acetic acid by 0.817.

Determination of Alcohol.—Alcohol is present in very small amounts in fruit vinegar that has not been completely acetified. Frear recommends concentrating the distillates as follows: Neutralize 100 cc. of the sample and distill off 40 cc. Then redistill the distillate till 20 cc. have gone over. Cool to 15.6° C. and make up to 20 cc. with distilled water. Determine the specific gravity with a 10-cc. pycnometer, and ascertain from the table on page 531 the per cent by weight of alcohol corresponding to the specific gravity. The percentage in the last distil-



to the mixture add a few drops of a solution of methyl violet (one part of methyl violet 2B in 100,000 parts of water). In the presence of mineral acids, a blue or green coloration will be produced.

Determination of Free Mineral Acids.—*Hehner's Method.**—To a weighed quantity of the sample add an excess of decinormal alkali, evaporate to dryness, incinerate, and titrate the ash with decinormal acid. The difference between the number of cubic centimeters of alkali added in the first place, and the number of cubic centimeters needed to titrate the ash, represents the equivalent of the free acid present.

Detection and Determination of Sulphuric Acid.—This is determined as barium sulphate by the addition of barium chloride solution. A slight cloudiness on the addition of the reagent indicates the presence of small quantities of sulphate as an impurity, rather than free sulphuric acid. If a minute quantity of free sulphuric acid be present, a rather heavy white cloud on the addition of the barium chloride will be formed, which slowly settles out. According to Brannt, if the quantity of sulphuric acid is more than one part in a thousand, the sulphate of barium formed by addition of the reagent produces a copious precipitate that rapidly falls to the bottom of the receptacle. This may be filtered, washed, ignited, and weighed in the usual manner.

Detection of Free Hydrochloric Acid.—Distill off half of a measured volume of vinegar into the receiving-flask of a distillation apparatus, and to the distillate add a few drops of nitrate of silver reagent. A precipitate indicates hydrochloric acid.

Detection of Malic Acid (*Free or Combined*).—Absence of malic acid may be assured, if no precipitate occurs with neutral acetate of lead, when a few drops of a solution of this reagent are added to the vinegar. In the presence of malic acid, as in the case of a pure cider vinegar, the precipitate which is formed with lead acetate is flocculent, forms at once, and is of considerable amount. In pure cider vinegar the precipitate will settle to the bottom of the test-tube, leaving a clear supernatant liquid within ten minutes. Unfortunately the acetate of lead test is a negative one, in that several organic acids other than malic will cause a precipitate, as, for instance, tartaric and saccharic acids, the former being found in wine and the latter in molasses vinegar. Malt vinegar also gives a copious precipitate with lead acetate, due to phosphoric acid.

The writer employs the following test† for detecting malic acid in

* Analyst, 1, 1877, p. 105.

† An. Rep. Mass. State Board of Health, 1902, p. 485. Food and Drug Reprint, p. 33.

vinegar: Add a few drops of a 10% solution of calcium chloride to some of the vinegar in a test-tube, and make the mixture slightly alkaline with ammonia. Filter off the precipitate that occurs at this point, to the filtrate add two or three volumes of 95% alcohol, and heat to boiling. A copious, flocculent precipitate of calcium malate will form, if malic acid be present, settling to the bottom of the tube in a few minutes. A precipitate will occur in malt and glucose vinegar, due to dextrin.

To confirm the presence of malic acid, filter, wash the precipitate with a little alcohol, dry, dissolve it in strong nitric acid in a porcelain evaporating-dish, and evaporate to dryness over the water-bath, forming calcium oxalate. Boil the residue with sodium carbonate, filter, acidify the filtrate with acetic acid, boil to expel the carbon dioxide, and add a solution of calcium sulphate. A precipitate of calcium oxalate confirms the presence of malic acid.

For the determination of malic acid proceed as directed on page 569.

Determination of Acid Tartrate of Potassium.—*Berthelot and Fleurien's Method.**—Twenty-five cc. of the vinegar are evaporated on the water-bath to syrupy consistency, and the residue is dissolved in water and made up to its original volume. It is then transferred to a 250-cc. Erlenmeyer flask, and 100 cc. of a mixture of equal parts of strong alcohol and ether are added, the flask is corked, shaken, and set on ice or in a cold place for forty-eight hours. At the end of this time, if a crystalline precipitate has gathered, the supernatant liquid is decanted upon a filter, and finally the precipitate is washed upon it by a fresh quantity of the ether-alcohol mixture, and the washing continued with this reagent till practically free from acid. The filter and its contents are then transferred to the original flask, and the tartrate is dissolved in boiling water,



slight effect on the polarization. In case of dark-colored or turbid samples, add to 50 cc. of the sample 5 cc. of about equal quantities of lead subacetate and alumina cream, shake, filter, and polarize in a 200-mm. tube, adding 10% to the reading on account of the dilution. The polarization value of the vinegar is conveniently expressed in terms of actual direct reading obtained by the undiluted sample in a 200- or 400-mm. tube.

If the invert reading is desired for calculation of sucrose or commercial glucose, subject the sample to inversion with hydrochloric acid and heat, as in the case of sugars.

For the determination of sucrose, use Clerget's formula (p. 483), calculating the true direct and invert readings from the direct and invert readings of the undiluted vinegar on the basis of the normal weight of the sample, by multiplying the obtained readings by 0.26 in the case of the Soleil-Ventzke instrument.

Determination of Reducing Sugars before and after Inversion.—Two portions of 25 cc. each are measured into 100-cc. flasks. One portion is diluted with 25 cc. of water, 5 cc. of concentrated hydrochloric acid are added, and the solution subjected to inversion by heating to 70° for 10 minutes and cooling. Both portions are neutralized with sodium hydroxide and made up to the mark. The reducing sugars are determined in each portion by Defren's modification of O'Sullivan's method (page 489) and calculated as dextrose.

Levulose may be determined as on page 508, polarizing the vinegar at two different temperatures.

ADULTERATION OF VINEGAR.

Standards of Purity.—In nearly all localities where pure-food laws prevail there are special provisions setting forth the requirements of pure vinegar as to percentage of acids, solids, and other conditions, differing considerably with the character of the vinegar used. Thus, in England, where the principal vinegar is malt vinegar, the legal standards are considerably different from those in force in France and Germany, where wine vinegar is prevalent. These differ again from the requirements found in the United States and Canada, where cider vinegar is the chief product.

Most of the state food laws fix a standard for the acidity of cider vinegar varying from 3.5 to 4.5 per cent of acetic acid, and in most cases also a minimum standard for total solids or residue of from 1.5 to 2 per

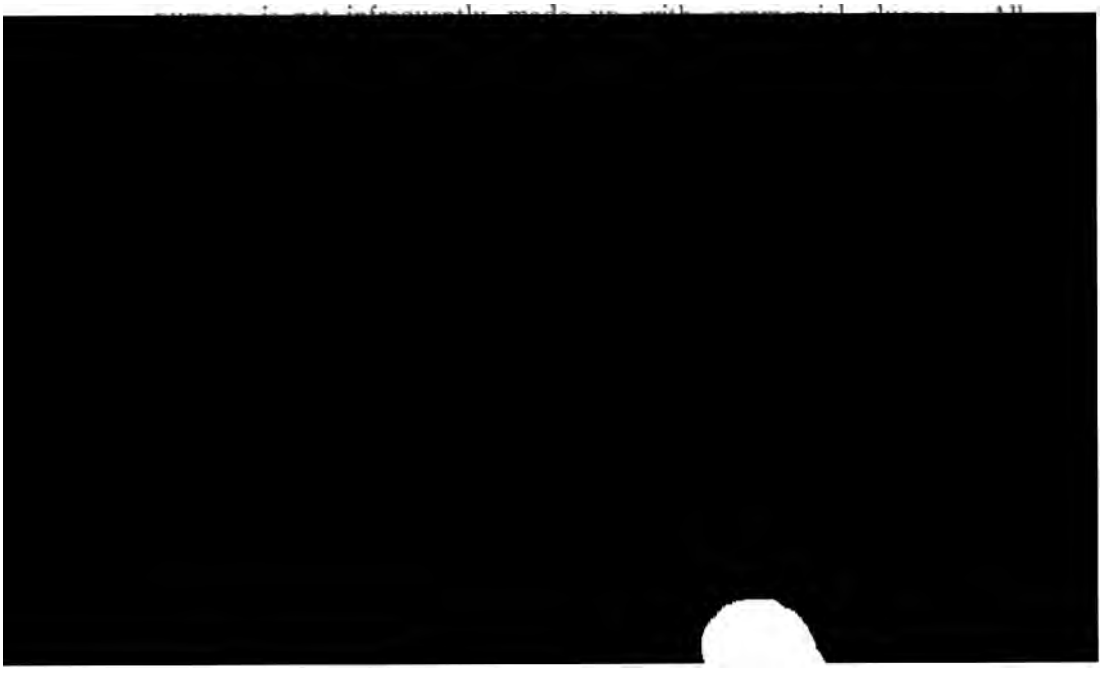
cent. Special laws stipulate furthermore in some states that cider vinegar, sold as such, must be *exclusively* the product of pure apple cider. In such cases cider vinegar may be adulterated by non-conformance to the standard in either acidity or solids or both, while yet it may be exclusively made from pure apple cider. This may be due either to actual watering or to incomplete acetification. On the other hand, so-called cider vinegar may be of legal standard as to solids and acidity, and yet be entirely spurious.

Accidental Adulteration of vinegar may result in the presence of injurious metallic salts, such as of copper, lead, or zinc, derived from vessels or utensils used in the manufacture of vinegar, or even minute traces of arsenic may be found, when glucose has been employed as an ingredient or source of the vinegar, the arsenic being in this case probably due to impure sulphuric acid used in the manufacture of the glucose.

Willful or Fraudulent Adulteration is, however, common, in which misbranded vinegar is sold under names suggesting a class other than that to which it really belongs, or wherein entirely artificial substitutes are made up for pure cider, malt, or wine vinegar, in which the color, residue, and acid principle may be either or all of spurious origin.

Artificial Cider Vinegar is in most cases readily detected, though very ingenious imitations are on the market, involving not a little skill and chemical knowledge in their manufacture.

Entirely artificial substitutes for cider vinegar are frequently made up of spirit vinegar, colored with caramel, and having the solids reinforced by apple jelly, made for the most part out of exhausted apple pomace, which is the residue left after the apple-stock has been subjected to one and sometimes two pressings. The jelly used for this



of molasses is very apparent in the residue of vinegar having sugar-house wastes, and the smell of a malt-vinegar residue is also very characteristic. If pyroligneous or wood vinegar has been introduced, the dried residue will have a tarry or smoky taste and smell.

The residue of cider vinegar is very soluble in alcohol, while that of malt vinegar is only slightly soluble. Wine vinegar residues dissolve readily in alcohol, except for the granular residue of cream of tartar. If the loop of a clean platinum wire be rubbed in the vinegar residue and ignited in a colorless Bunsen flame, the color imparted will, if the vinegar has been made from pure cider exclusively, consist altogether of the pale-lilac color of a potash salt without any of the yellow sodium flame being visible. In all vinegars other than of pure cider, the sodium flame will predominate, when the residue is burnt as above. Again, the ignited residue left in the loop of wire in the case of a pure cider vinegar will form a fusible bead, having a strong alkaline reaction upon moistened test-paper, and effervescing briskly when immersed in acid. The presence in vinegar of even a slight trace of added mineral acid will prevent the ignited residue from having the alkaline reaction, or effervescing with acid.*

The residue of malt or beer vinegar is brown and gummy, containing a considerable quantity of dextrin. Not only are the appearance and odor of the dried vinegar residue to be particularly noted, but also the odor given off in the first stages of burning this residue to an ash. With cider vinegar the apple odor is very marked while burning. In vinegar wherein molasses products have been employed, the smell of charred sugar is usually apparent, while with glucose vinegar the smell of burnt corn predominates.

On burning the residue of malt vinegar, the odor produced at first is not unlike that of toasted bread. At a later stage in the burning the vapors evolved are very pungent.

The Character of the Ash is of considerable importance in determining the source of a sample of vinegar. The ash of pure cider and malt vinegar is quite strongly alkaline, while that of distilled and wood vinegar is only slightly alkaline. The ash of cider vinegar is high in alkaline carbonates.

In cider and malt vinegar the quantity of phosphoric acid present in the ash is considerable, while only traces are present in distilled or spirit vinegar. Considerably more than half the phosphoric acid in

* Davenport, 18th An. Rep., Mass. Board of Health, 1887, p. 159.

the ash of cider vinegar is soluble, while no soluble phosphoric acid is present in the ash of spirit vinegar.

The percentage of ash in total solids is of some value in judging the purity of cider vinegar. According to Frear,* if the ash of the vinegar is less than 10% of the total solids, the vinegar may be suspected of having added unfermented material, while a percentage of ash less than 6 is absolute evidence that the vinegar is not genuine cider vinegar.

The alkalinity of 1 gram of the ash of pure cider vinegar should be equivalent to at least 65 cc. of tenth-normal acid. At least 50% of the phosphates in the ash should be soluble in water.

Character of the Sugars.—One of the most important steps in establishing the source of a vinegar consists in subjecting it to polarization (p. 618). From the nature of the sugar-content of the apple juice, not only when freshly expressed, but also when allowed to undergo alcoholic fermentation, and, furthermore, after it has gone over into vinegar, the polarization through all three stages is *always* left-handed.

Browne † has shown that the optical rotation of the freshly expressed juice of eleven varieties of apple varies from 19.24° to 49° to the left on the Ventzke scale, in a 400-mm. tube. Also that in the case of five samples of completely fermented cider, examined five or six months after pressing, the left-handed rotation in a 400-mm. tube varied from 1.76° to 5.28°. He showed, furthermore, that a sample of pure cider jelly made up of concentrated apple juice had a left-handed rotation amounting to 21.35° in a 200-mm. tube (20 grams made up 100 cc.), and finally that four cider vinegar samples of known purity showed left-handed readings of from 0.96° to 2.94° Ventzke in a 400-mm. tube.

The left-handed rotation of pure cider vinegar is a characteristic so fixed and unalterable that a right-handed polarization of more than 0.5°

Frear regards the ratio of reducing sugars after inversion to total solids as a useful factor in discriminating between pure cider vinegar and the common artificial substitutes in which the solids of distilled vinegar are reinforced by apple jelly, or in which commercial glucose or molasses vinegars are used. When the reducing sugars after inversion form more than 25% of the entire solids, the alleged cider vinegar is undoubtedly spurious. In pure cider vinegar the per cent of reducing sugar is the same after inversion as before. The same is true of glucose vinegar.

Vinegar containing added molasses or cane sugar will, however, naturally show an increase in reducing sugar after inversion.

A large content of alcohol in cider vinegar, otherwise showing the constants of pure vinegar except for the low acidity, would indicate incomplete acetification. A high content of nitrogen is characteristic of malt vinegar.

Data of analyses of samples of vinegar examined in the Food and Drug Department of the Massachusetts State Board of Health are given in the tables on this page and the next. The table below shows in summarized form the results obtained from the examination of eighty-four samples of undoubtedly pure cider vinegar examined in 1901.*

CIDER VINEGAR FOUND PURE.

	Acid (Per Cent).	Solids (Per Cent).	Ash (Per Cent).	Polarization.
Maximum	6.36	4.00	0.58	−5.4
Minimum	4.50	2.01	0.19	−0.4
Mean	4.84	2.43	0.38	−2.0

The second table includes samples of adulterated vinegar, sold for cider vinegar, none of which were probably made from cider. It will be noticed that in several of the samples the amount of glucose was abnormally large, as is shown by the very high right-handed polarization, in one case amounting to over 12°.

Direct Tests Made on the Vinegar.—The genuine or spurious nature of cider vinegar may usually be established by direct tests with reagents on the vinegar itself. The appearance, taste, and odor of the vinegar should be noted. Brannt † applies the test of odor in vinegar as deter-

* 32d An. Rep. (1900), p. 661, Food and Drug Reprint, p. 44; 33d An. Rep. (1901), p. 467, Food and Drug Reprint, p. 47; 34th An. Rep. (1902), p. 483, Food and Drug Reprint, p. 31.

† A Practical Treatise on the Manufacture of Vinegar, p. 219.

VINEGAR NOT THE EXCLUSIVE PRODUCT OF PURE APPLE CIDER.

Per Cent Acetic Acid.	Per Cent Total Solids.	Per Cent Ash.	Per Cent Ash in Total Solids.	Polarization in 200-mm. Tube.	Lead Acetate.
5.90	.40	....	....	+1.4	No precipitate
5.14	.36	....	....	.0	" "
5.12	.53	....	....	+ .6	" "
4.83	3.70	.32	8.65	+8.0‡	" "
4.82	2.71	.13	4.80	+9.6‡	Heavy precipitate*
4.80	1.97	.20	10.15	+ .9	Precipitate
4.80	1.03	.27	14.75	+1.1	"
4.66	2.92	.20	6.49	+2.2	No precipitate
4.60	2.57	....	....	+2.6	" "
4.56	2.60	....	....	+7.0‡	†
4.54	3.97	.19	4.78	+5.6	No precipitate
4.54	3.90	.32	9.72	+5.0	" "
4.54	2.94	.23	7.82	+5.0	" "
4.54	2.70	.23	8.52	+ .4	Precipitate
4.50	3.05	....	....	+2.2	No precipitate
4.50	2.92	.22	7.52	+ .9	" "
4.50	2.69	....	....	+2.8	" "
4.48	3.80	....	....	+12.0‡	" "
4.46	2.80	....	....	+2.6	" "
4.42	2.75	....	....	+3.2	Slight precipitate
4.42	2.10	....	....	+9.2	Precipitate
4.40	2.51	.20	11.15	+1.1	"
4.40	.97	....	....	+ .4	No precipitate
4.38	.29	....	....	+1.6	" "
4.32	.70	.09	12.86	....	" "
4.08	3.35	....	....	+1.2	Precipitate
3.98	.55	....	....	+1.8	Slight precipitate

* Cider vinegar to which apple jelly containing glucose had been added for the purpose of increasing the solids after watering.

† This sample contained a large amount of phosphate, and consequently the test for malates is obscured.

‡ These samples polarized practically the same after as before inversion, indicating much glucose.

mining its character, by rinsing out a large beaker with the sample, and, after allowing it to stand for some hours, examining the few drops remaining in the beaker. The acetic acid having for the most part become

The character of the precipitate produced by neutral lead acetate should be particularly noted. Unless it is flocculent and copious, settling out after a few minutes, cider vinegar is not pure, even if a marked turbidity is produced. Added apple jelly from exhausted apple pomace gives such a turbidity, and is to be suspected when not more than a cloudiness is produced on addition of the lead acetate reagent. Pure cider vinegar should respond in a perfectly normal manner to both the lead acetate and the calcium chloride tests for malic acid.

Wood Vinegar or Pyroligneous Acid is sometimes rendered apparent by the empyreumatic or tarry taste and odor imparted to the product. When, however, the added acetic acid has been so purified that the tarry taste and odor are lacking, its presence may often be proved by the traces of furfurol which always accompany it.

Test for Furfurol.—A little of the vinegar is subjected to distillation, and to the first few drops of the distillate is added a little colorless anilin solution. A fading crimson color will be produced in presence of furfurol. This reaction may sometimes be obtained upon the vinegar itself without distillation, if sufficient added wood vinegar be present.

The first portion of the distillate of wood vinegar reduces permanganate of potassium to a marked degree.

The Addition of Spices to vinegar in order to increase the pungency is best detected by first neutralizing the vinegar with sodium carbonate and then tasting. Under these conditions, the admixture of spices is rendered very apparent.

Detection of Caramel.—Considerable added caramel in vinegar is apparent from the unnaturally dark color and extremely bitter taste of the residue after evaporation.

Tests for caramel made on the vinegar residue, if long dried at the temperature of the water-bath, are not to be depended on as establishing the presence of added caramel, since at that temperature the decomposition of the sugar may in any event cause a positive test.

Caramel in vinegar is best detected by the Crampton and Simons' method with fullers' earth, as described on page 603. Other methods are given in Chapter XVI.

Examination for Metallic Impurities.—*Lead and Zinc* are best looked for in the ash of the vinegar in cases where, like cider vinegar, the percentage of extract is high. A large volume of the vinegar is evaporated to substantial dryness over the water-bath. This may most readily be done in a 100-cc. platinum wine-shell, adding the vinegar in successive

portions. To the residue add a small amount of sodium hydroxide, and burn to an ash in a muffle, or over a low flame, using potassium nitrate if necessary, a little at a time. Take up the ash in dilute hydrochloric acid, and examine for lead and zinc as in the case of canned goods.

In the case of vinegar low in extract, as in spirit vinegar, the sample may be evaporated to dryness, the residue dissolved directly in dilute hydrochloric acid without ignition, and the acid solution subjected to direct examination for lead and zinc.

Copper is best determined by electrolysis. 100 cc. of the vinegar are evaporated to a volume of about 10 cc. with a little sulphuric acid, filtered into a platinum dish, and subjected to electrolysis, using conveniently the apparatus described on page 493.

Arsenic.—Boil down a portion of the vinegar, to which concentrated nitric acid has been added, to a small volume, then add a few cubic centimeters of concentrated sulphuric acid, and continue the heating till fumes of sulphuric acid show the nitric to have been driven off. Cool, dilute with water, and test in the Marsh apparatus.

REFERENCES ON VINEGAR.

ALLEN, A. H. White Wine Vinegar. Analyst, 21, 1896, p. 253.

ALLEN, A. H., and MOOR, C. G. Vinegar. Analyst, 18, 1893, pp. 180 and 240.

BERSCH, J. Die Essigfabrikation. Vienna, 1895.

BRANNT, W. Vinegar, Acetates, Cider, Fruit Wines and Preservation of Fruits. London, 1900.

BROWNE, C. A. A Chemical Study of the Apple and Its Products. Penn. Dept. of Agric. Bul. 58, 1899.

CRAMPTON, C. A., and SIMONS, F. D. Detection of Caramel in Spirits and Vinegar Jour. Am. Chem. Soc., 21, 1899, p. 355.

SANGLÉ-FERRIÈRE. Vinaigre. Analyse des Matières Alimentaires (Girard et Dupré), p. 237.
SMITH, A. W. Vinegar Analysis and Characteristics of Pure Cider Vinegar. Jour. Am. Chem. Soc., 20, 1898, p. 3.
SYKES, W. J. Detection of Adulteration in Vinegar. Analyst, 16, 1891, p. 83.

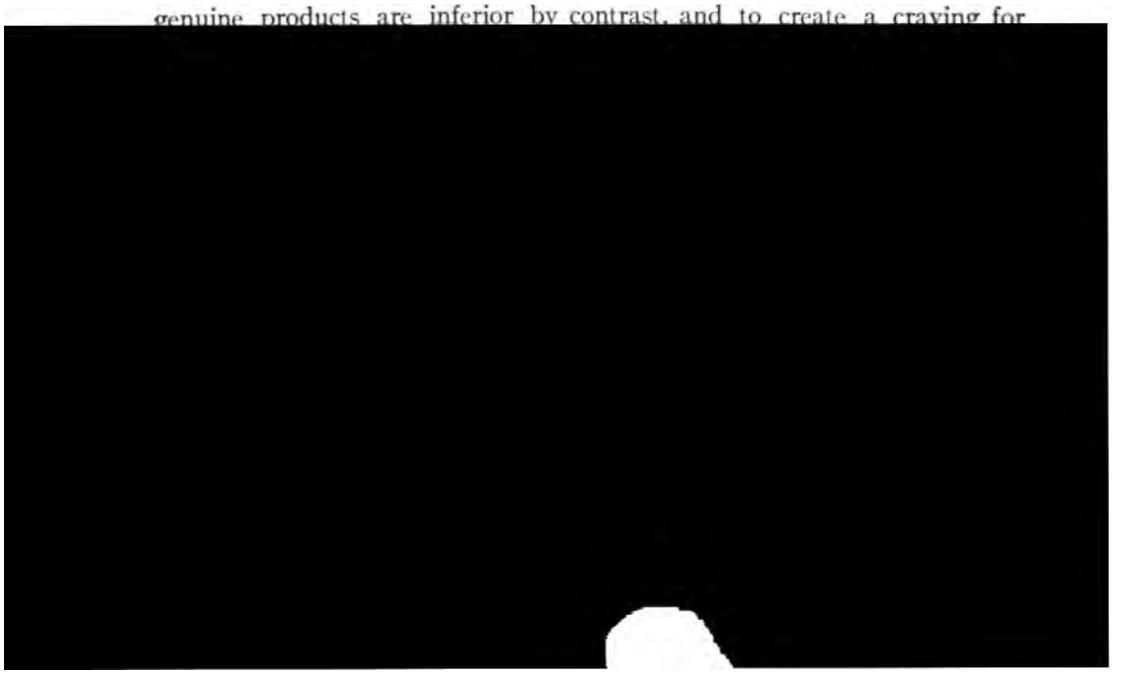
Connecticut Exp. Sta. An. Reports, 1897, 1898, 1899.
Massachusetts State Board of Health, An. Reports, 1900, 1901, 1902, and 1903.
North Carolina Exp. Station Bul. 153.

CHAPTER XVI.

ARTIFICIAL FOOD COLORS.

THE use of artificial dyestuffs in food products has greatly increased during the last decade, both in degree and in variety of colors employed. Where formerly but a few well-known coloring matters, chiefly so-called vegetable colors and occasionally mineral pigments were used for this purpose, a vast array of dyes, chosen largely from the coal-tar colors, are now found in food, so that at present the exact identification of the particular dyestuff employed in all cases presents a somewhat formidable problem to the analyst. The problem may consist in determining the class to which a commercial food color or combination of colors belongs, or it may consist in isolating the color itself, and afterwards identifying it as far as possible, for the purpose of determining whether or not it is harmless within the meaning of the law.

The effect of imparting to the cheaper varieties of jellies, jams, and ketchups which flood the market such intense and striking colors that these products in no wise resemble their pure uncolored prototypes, has a tendency in many cases to mislead the public into the idea that the genuine products are inferior by contrast, and to create a craving for



appear of greater value than they really are. In most states the food laws regarding employment of colors are so framed, that the presence of such colors constitutes an offense under one or the other of the above heads, mainly, however, because, by reason of their use, cheaper or inferior materials are made to masquerade for the higher or genuine grades, as, for instance, when alleged currant jelly is found to consist chiefly of apple-stock and commercial glucose, colored with an artificial red dye.

In such cases the analyst has merely to prove conclusively that an artificial color is present, even if he does not identify the dye itself. It is of course more satisfactory to at least show in addition whether the dye present is of vegetable origin, or is of the coal-tar variety, and in most cases this can readily be done, even if it is not easy to identify the exact color.

In localities where laws prevail stipulating that what are commonly known as "mixtures" or "compounds" to be legally sold, must be labeled with the names and percentages of ingredients, the law applies to coloring matters as well as other ingredients, and the exact dye or dyes employed should appear on the label. Otherwise the product must be classed as adulterated.

Toxic Effects of Colors.—Formerly the use of such pigments as chromate of lead was common in coloring confectionery, but lead chromate is rarely used at present. Other mineral pigments obviously unfit for use in food by reason of their well-known poisonous effects are those which contain salts of arsenic, mercury, lead, and copper. While most of the coal-tar colors are considered harmless in themselves, some are decidedly objectionable, and should not be used in foods. Under the latter class are included, first, those in connection with the manufacture of which arsenic, mercury, or other poisonous mineral ingredients have been used, such for example as arsenical fuchsin, and, second, those which are themselves inherently poisonous, as for instance picric acid. Fuchsin is now largely made without the aid of arsenic acid, and this variety is entirely harmless. The toxic effects of many of the coal-tar colors have not been thoroughly established excepting in a negative way. Weyl has made many experiments on dogs and rabbits in which these animals have been fed with varying amounts of coloring material. In nearly all cases the doses far exceeded the amounts ordinarily taken in food, and the experiments are of value mainly in so far as they show harmless results of certain colors on the animal. It is to be regretted

that physiological experiments cannot more readily be tried on human beings, so as to study the effects of administering to them such amounts as are used in food.

More conclusive results (though still of a negative character) tending to establish the harmlessness of most of the coal-tar colors are given by Grandhomme * in statistics showing the condition of health of laborers in factories where these dyestuffs are made, in comparison with those engaged in other industries where poisonous materials are handled. From these it appears that the proportion of illness among the anilin-makers is remarkably small.

In the case of coloring confectionery by the use of mineral pigments, a considerable amount of the coloring material must be used, forming without doubt a source of danger in some cases. With coal-tar dyes, on the contrary, the case is different. One ounce of auramine, for instance, has been found sufficient to give a deep-yellow color to 2,000 pounds of confectionery, so that almost an infinitesimal amount of the actual dyestuff is taken into the system. Hence it is that very little danger need be apprehended from the use of most coal-tar colors in food, objectionable as they certainly are as a commercial fraud.

Injurious and Non-injurious Colors.—Various countries have enacted specific laws regulating the use of coloring matters in foods, especially England, France, Germany, Austria, and Italy. In some cases attempts have been made to specify harmful and harmless colors. The National Confectioners' Association of the United States has compiled a useful classified list of injurious and harmless colors,† the classification being based largely on the results of experiments by Weyl and König, as well as upon the Resolutions of the Association of Swiss Chemists, and on the French Ordinances regarding food colors. The list is as follows:



Compounds of Mercury.—Vermilion, etc.

Compounds of Arsenic.—Scheele's green, Schweinfurth green, etc.

In Other Words colors in whose preparation mercury, lead, copper, arsenic, antimony, tin, zinc, chromium, and barium compounds are used.

HARMFUL ORGANIC COLORS.

Red Colors.—*Ponceau 3RB.*—Ponceau B extra, fast ponceau B, new red L, scarlet EC, imperial scarlet, old scarlet, Biebrich scarlet.

Croccin Scarlet 3B.—Ponceau 4RB.

Cochenille Red A.—Crocein scarlet 4B and G, brilliant scarlet, brilliant ponceau 4R, ponceau 4R, ponceau brilliant 4R, new coccin, scarlet.

Crocein Scarlet 7B.—Crocein scarlet 8B, ponceau 6RB.

Crocein scarlet O extra.

Safranin.—Safranin T, safranin extra G, safranin G extra GGSS, safranin GOOO, safranin FF extra No. O, safranin conc., safranin AG extra, safranin AGT extra, anilin pink.

Yellow Colors.—***Gum gutta.***

Picric acid.

Martius Yellow.—Naphthylamin yellow, jaune d'or, Manchester yellow, naphthalin yellow, naphthol yellow, jaune naphthol.

Acme Yellow.—Chrysoin, chryscolin yellow T, gold yellow, resorcin yellow, acid yellow RS, tropæolin O, jaune II.

Victoria Yellow.—Victoria orange, anilin orange, dinitrocresol, saffron substitute, golden yellow.

Orange II.—Orange No. 2, orange P, orange extra, orange A, orange G, acid orange, gold orange, mandarin G extra, beta-naphtholorange, tropæolin OOO No. 2, mandarin, chrysaurin.

Metanil Yellow.—Orange MN, tropæolin G, Victoria yellow (O double conc.), jaune G (metanil extra).

Sudan I.—Carminnaphte.

Orange IV.—Orange No. 4, orange N, orange GS, new yellow, acid yellow D, tropæolin OO, fast yellow, diphenylorange, diphenylamine orange, jaune d'anilin, anilin yellow.

Green Colors.—*Naphthol green B.*

Blue Colors.—*Methylene blue BBG.*—Methylene blue BB, in powder extra, methylene blue DBB extra, methylene blue BB (crystalline) ethylene blue.

Brown Colors.—*Bismarck Brown.*—Bismarck brown G, Manchester brown, phenylen brown, vesuvin, anilin brown, leather brown, cinnamon brown, canelle, English brown, gold brown.

Vesuvin B.—Manchester brown EE, Manchester brown PS, Bismarck brown, Bismarck brown T, brun Bismarck EE.

Fast Brown G.—Acid brown.

Chrysoidin.—Chrysoidin G, chrysoidin R, chrysoidin J, chrysoidin Y.

HARMLESS MINERAL COLORS.

Blue Colors.—*Ultramarine blue.*

Violet Colors.—*Ultramarine violet.*

Brown Colors.—*Manganese brown.*

Chocolate-brown and colors of a similar nature have as their basis natural or precipitated oxide of iron, which in an impure condition may have small quantities of arsenic in its composition. It is possible with proper care to secure a raw material entirely free from this objectionable element, and no oxide of iron containing any traces of arsenic should be used in the preparation of color.

Green Colors.—*Ultramarine green.*

HARMLESS ORGANIC COLORS.

Red Colors.—*Cochineal carmine.*

Carthamic acid (from saffron).

Redwood.

Artificial alizarin and purpurin.

Cherry and beet juices.



Bordeaux B.—Fast red B, Bordeaux R extra.

Cerasin.—Rouge B.

Ponceau 2G.—Brilliant ponceau GG, ponceau JJ.

Fuchsin S.—Acid magenta, rubin S, fuchsin acide (free from arsenic).

Archil Substitute.—Naphthion red.

Orange I.—Orange No. 1, naphtholorange, alpha-naphtholorange, tropæolin OOO No. 1.

Congo red.

Azorubin S.—Azorubin, azorubin A, azoacidrubin, fast red C, carmoisin, brilliant carmoisin O, rouge rubin A.

Fast Red D.—Fast red EB, fast red NS, amaranth, azoacidrubin 2B, Bordeaux DH, Bordeaux S, naphthol red S, naphthol red O, Victoria ruby, wool red (extra), œnanthinin.

Fast Red.—Fast red E, fast red S, acid carmoisin S.

Ponceau 4GB.—Crocein orange, brilliant orange G, orange GRX, pyrotin orange, orange ENL.

Fuchsin.

Metanitrazotin.

Yellow and Orange Colors.—*Annatto.*

Saffron.

Safflower.

Turmeric.

Naphthol Yellow S.—Citronin A, sulphur yellow S, jaune acide, jaune acide C, anilin yellow, succinine, saffron-yellow, solid yellow, acid yellow S.

Brilliant Yellow.—(Sch.)

Ponceau 4GB.—Crocein orange, brilliant orange G, orange GRX, pyrotin orange, orange ENL.

Fast Yellow.—Fast yellow G, fast yellow (greenish), fast yellow S, acid yellow, new yellow L.

Fast Yellow R.—Fast yellow, yellow W.

Azarin S.

Orange I.—Orange No. 1, naphtholorange, alpha-naphtholorange, tropæolin OOO No. 1.

Orange.—Orange GT, orange RN, brilliant orange O, orange N.

Mixtures of harmless red and yellow colors.

Green Colors.—*Spinach green.*

Chinese green.

Malachite Green.—Malachite green B, benzaldehyde green, new Vic-

toria green, new green, solid green crystals, solid green O, diamond green, bitter amond oil green, fast green.

Dinitrosoresorcin.—Solid green O in paste, dark green, chlorine green, Russia green, Alsace green, fast green, resorcinol green.

Mixtures of harmless blue and yellow colors.

Blue Colors.—*Indigo.*

Litmus.

Archil blue.

Gentian Blue 6B.—Spirit blue, spirit blue FCS, opal blue, blue lumière, Hessian blue, light blue.

Coupiers Blue.—Fast blue R and B, solid blue RR and B, indigin DF, indulin (soluble in alcohol), indophenin extra, blue CB (soluble in alcohol), nigrosin (soluble in alcohol), noir CNN.

In General such blues as are derived from triphenylrosanilin or from diphenylamin.

Violet Colors.—*Paris Violet.*—Methyl violet B and 2B, methyl violet V_3, pyoktanin cœruleum, malbery blue.

Wool black.

Naphthol black P.

Azoblue.

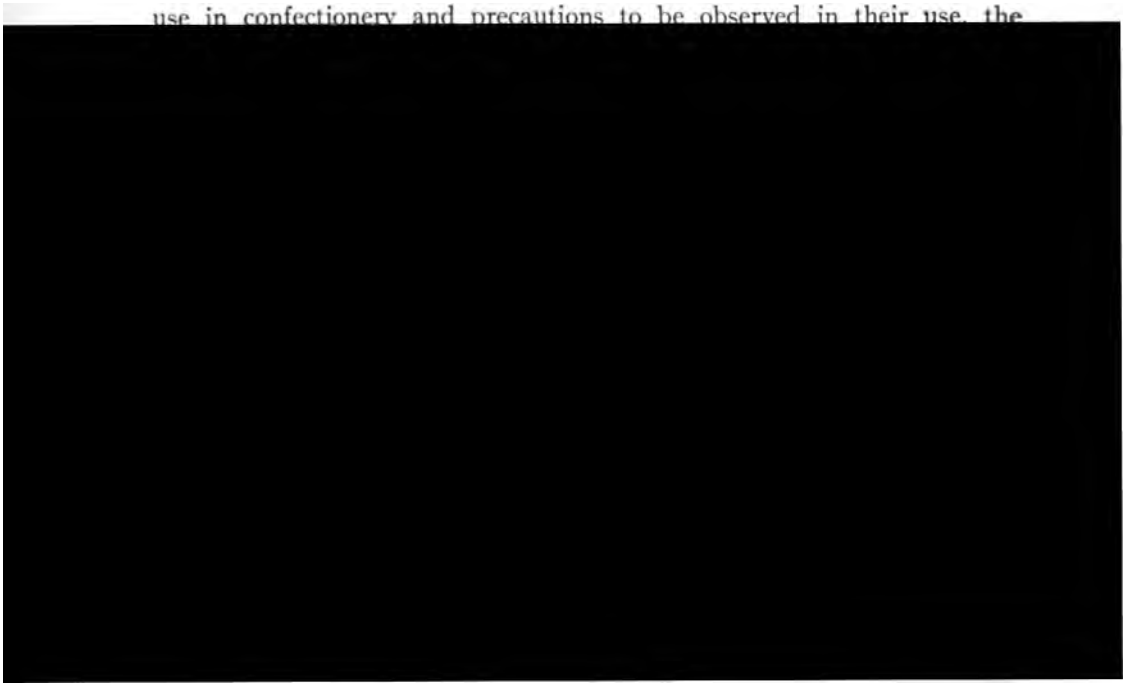
Mauvein.—Rosolan, violet paste, chrome violet, anilin violet, anilin purple, Perkins violet, indisin, phenamin, purpurin, tyralin, tyrian purple, lydin.

Brown Colors.—*Caramel.*

Licorice.

Chrysamin R.

Use of Colors in Confectionery.—Regarding the choice of colors for use in confectionery and precautions to be observed in their use, the



fectioners are in duty bound to provide against all possible contingencies of harm, by using the utmost care in obtaining absolutely non-poisonous colors, buying only from color-dealers of established reputation and unquestioned responsibility, whose colors are tested at frequent intervals, and are vouched for by competent chemists.

Confectioners should require that a guarantee be put upon each package of color, stating not only that the contents are non-poisonous, but also that they will not in any way interfere with digestion or injure health.

Fourth. Any *illegitimate* use of coloring matter in confectionery as a substitute for chocolate or any other material or ingredient, or for the purpose of adding bulk or increasing the weight of the confectionery in which it is incorporated, should not be permitted or countenanced. Both the letter and the spirit of these laws should clearly prevent the *illegitimate* use of coal-tar colors or of earth colors, such, for example, as chocolate-brown, coconole brown, or chocolatina.

Fifth. That color-dealers furnishing colors to confectioners should publish printed lists of their colors under the various names and titles by which they are known and offered for sale, accompanying such lists with ample certifications by competent chemists to their purity and suitableness for coloring confectionery and other articles of food. They should also attach to each package or other container of color a guarantee that it does not contain anything injurious to health.

VEGETABLE COLORS.

These with a few mineral pigments and cochineal were formerly almost exclusively used for coloring food products, and are still used to some extent.

Most of the vegetable colors, according to L. Robin,* react with ammonia to form a coloration, usually passing from violet to blue, then to a brownish green, when the ammonia is added little by little in excess to the color in solution. If by the addition of ammonia to a solution of an unknown color the green coloration does not result, the presence may be suspected of orchil or cudbear, logwood, cochineal, or a coal-tar dye.

The following vegetable colors are occasionally found in food, with some of the reactions in aqueous solution, as given by Robin:

* Girard et Dupré, Analyse des Matières Alimentaires, p. 579.

RED COLORS.

Nature of Color.	Ammonia.	Alum and Sodium Carbonate 20% Solution.		Mixture of Aluminum Acetate and Sodium Carbonate.
		Lake.	Filtrate from	
Bilberry (whortleberry)	Dull greenish	Greenish blue, rose-colored on edges	.....	Bluish violet
Beet.	Muddy yellow, brown, or rose-red	Dull green or rose	.....	Garnet
Black currant.	Deep green	Greenish blue	Bottle-green	Violet-blue
Logwood.	Red tinged with violet	Blue tinged with violet	.....	Tinged with violet
Brazil wood.	Currant-red	Rose	Rose tinged	Lilac to wine color
Raspberry.	Bluish green	Rose tinged	Bluish green	Lilac tinged with violet
Currant.	Yellow-brown to greenish	Gray to lilac	Dull maroon to bottle-green	Red-maroon
Blackberry	Yellowish green	White or rose violet	Bluish	Dull violet
Phytolacca.	Lilac	Violet	.....	Clear violet, passing to yellow with ammonia
Elderberry.	Light green	Blue tinged with violet	.....	Violet, quickly passing to blue with acetate of copper

YELLOW COLORS.

Nature of Color.	Ammonia.	Hydrochloric Acid.	Alum and Carbonate of Soda 20% Solution. Lake.
Persian berries.	Yellow-red	Precipitate yellow-brown	Orange
Old fustic.	Very bright yellow	Yellow-orange	Orange
Quercitron bark	Becomes clearer	Bright yellow pre-	Yellow-red tending to

REACTIONS OF COLORING MATTERS.

Coloring Matter.	Concentrated H_2SO_4.	Concentrated HNO_3.	$H_2SO_4 + HNO_3$.	Concentrated HCl.
Annatto....	Indigo-blue, changing to violet	Blue, becoming colorless on standing	Same	No change, or only slight dirty yellow and brown
Turmeric...	Pure violet	Violet	Violet	Violet, changing to original color on evaporation of HCl
Saffron.....	Violet to cobalt blue, changing to reddish brown	Light blue, changing to light reddish brown	Same	Yellow, changing to dirty yellow
Carrot.....	Umber brown	Decolorized	Same with NO_2 fumes and odor of burnt sugar	No change
Marigold...	Dark olive-green, permanent	Blue, changing instantly to dirty yellow-green	Green	Green to yellowish green
Safflower...	Light brown	Partially decolorized	Decolorized	No change

of aluminum acetate or potassium bichromate) by boiling the mordanted fibers in a bath of the colored solution, rendered acid by acetic acid. The dyed fibers are then examined by reagents, as in tables given on pages 652–659.

SPECIAL TESTS FOR VEGETABLE COLORS.—*Orchil* and *Cudbear*, both derived from lichens, dye wool red in acid bath. The colored fiber, in the case of cudbear, is turned blue by treatment with ammonia. For reactions of orchil on the fiber see table, page 654. Robin's test for orchil in aqueous solution consists in shaking it with ether, which, if orchil is present, is colored yellow. On treatment of the ether with ammonia, the yellow color is changed to blue, and, by adding acetic acid, goes over to a reddish violet.

Logwood, according to Robin, in aqueous solution colors ether yellow, and on treating the ether with ammonia the color becomes red or faintly violet. Potassium bichromate gives a violet coloration, mingled with greenish yellow. If cotton is first mordanted by boiling with aluminum acetate, it is dyed violet when boiled in a solution of logwood.

Turmeric is best extracted from a dry residue with alcohol, which it colors yellow. The color is transferred to a piece of filter-paper by soaking the paper in the alcoholic tincture, the paper is dried and dipped in a dilute solution of boric acid or borax slightly acidulated with hydrochloric acid. On again drying the paper, it will be of a cherry-red color if turmeric is present, and when touched with a drop of dilute alkali will turn dark olive.

Caramel.—Care should be taken in testing for caramel not to subject the sample to long-continued heating, even on the water-bath. Indeed caramel is sometimes developed spontaneously in saccharine food products during their process of manufacture when heat is used, by the charring of the sugar. If solutions are to be concentrated or brought to dryness before testing for caramel, this should be done in a vacuum desiccator over sulphuric acid, or at a temperature not exceeding 70°. For detection of caramel in milk, vinegar, and liquors, special tests are given elsewhere.

*Fradiss Test.**—The dried residue of the sample to be tested is extracted with warm, pure methyl alcohol, which, if caramel be present, is colored brown. Filter, and to the filtrate add amyl alcohol or chloroform. In presence of caramel, a brown flocculent precipitate is formed, which slowly settles to the bottom of the tube.

Indigo in aqueous solution turns green with ammonia. On boiling, the solution becomes bright blue. Indigo in neutral or acid solution dyes wool or silk.

MINERAL PIGMENTS.

Evidence of the presence of these pigments is usually best looked for in the ash of the suspected sample. In some cases the color may be extracted from the dried residue by water, alkali, or alcohol.

Prussian Blue.—This pigment is insoluble in water. It is decomposed and decolorized by treatment with potassium hydroxide. If the filtered alkaline solution of the coloring matter be treated with hydrochloric acid and ferric chloride, a precipitate of the original Prussian blue will be produced. For reactions on the fiber see table, page 658.

Ultramarine Blue is decolorized by hydrochloric acid. Treatment of

imparts to this solvent a yellowish color, the depth depending on the amount present. The separated amyl alcohol is washed with water till neutral, and divided into two portions. To one of these a little water is added, and then drop by drop a solution of uranium acetate, shaking each time a drop is added. In presence of cochineal the water is colored a very characteristic emerald-green color. To the other portion ammonia is added. If cochineal has been used, a violet coloration is produced. These reactions are very delicate.

COAL-TAR COLORS.

So many of the coal-tar dyes are adapted for use in food that it would be impossible to even name them all, especially in view of the fact that new colors are from time to time being added to the list. No attempt will be made in the present work to give the nature and composition of the dyes named, as such descriptions would lead beyond its scope. For detailed information along this line the reader is directed to the references on page 660, and especially to the works of Schultz and Julius, Benedict and Knecht, Weyl, etc.

About 2000 separate coal-tar dyes are at present on the market. Various classifications of these colors are attempted, based on (1), their origin, as ***anilin dyes***, ***naphthalin dyes***, ***anthracene dyes***, etc.; (2), their chemical composition, as ***nitro***, ***nitroso***, ***azo***, ***diazo***, and other compounds; (3), their solubility in water and other solvents; and (4), their mode of application to the fiber, as ***basic dyes***, ***acid dyes***, *direct cotton dyes*, *mordant dyes*, etc.

These dyes are sold in the form of powder, and are readily made into solutions for food colors in the case of the water-soluble varieties, and into pastes in the case of the insoluble forms. Most of the coal-tar colors employed in foods are naturally of the soluble variety, especially such as are found in jellies, jams, fruit products, canned foods, ketchups, beverages, and milk. Pastes made from insoluble dyes are adapted mainly for exterior coatings of hard substances such as candies. Colors in the dry form are to be looked for in such spices as cayenne, mustard, and mace, but a commoner method of coloring these spices high in oil is to mix with them a solution of the color in oil (usually cottonseed). Oil solutions of coal-tar dyes are also employed for coloring butter and oleomargarine.

The chief concern of the food analyst, as regards artificial color is

its recognition in food products. Coal-tar dyes may usually be identified as such, but it is not always possible to name the particular individual dye or combination of dyes employed, even though the class to which they belong may be determined. One reason for this is that not infrequently mixtures of two or more colors are employed.

DETECTION OF COAL-TAR COLORS IN FOODS.—There are various methods for the separation of coloring matters from food products, and these may be divided into three general classes: First, dying silk or wool with the color by boiling the fiber in a solution of the sample to be examined; second, extracting the color from a solution of the sample by the use of an immiscible solvent; and third, extracting the color from the dried residue of the sample by means of a suitable solvent. Of these the method of dying wool lends itself most readily to the analyst's use, by reason of its simplicity, and from the fact that almost without exception coal-tar dyes adaptable for food colors are substantive dyes, being readily taken up by wool.

Basic and Acid Dyes.—The soluble coal-tar dyes are either basic or acid. Basic dyes are precipitated from their aqueous solution by tannin. Acid dyes are not so precipitated. Theoretically, all the basic colors are taken up by wool from a faintly alkaline or neutral bath, while the acid colors are left in solution. Thus if a dilute solution of the color be made faintly alkaline with ammonia and boiled with the wool, only basic colors will be taken up. If both acid and basic dyes are present in the same solution, the basic color should first be exhausted by the use of fresh pieces of wool in the ammoniacal solution, till they no longer take out color, after which the solution should be made slightly acid with hydrochloric acid and again boiled with wool, which under these



be freed from grease by boiling first in very dilute soda solution and finally in water. Strips of the woolen cloth, or pieces of the worsted thus previously cleansed, are boiled in diluted filtered solutions of jams, jellies, ketchup, fruit and vegetable products, and similar food preparations, or in solutions of candy colors, or in wines, the clear solution of the sample to be tested being slightly acidified with hydrochloric acid.

Arata * recommends boiling the wool in a dilute solution of the food material to which potassium bisulphate has been added, using 10 cc. of a 10% solution of the bisulphate to 100 cc. of the solution to be tested. If the color solution is neutral, the wool should first be boiled in this before acidifying, to separate out any basic dyes. The dyed wool, after removal from the solution, is boiled first in water, and afterwards preferably in an alkali-free soap solution. It is then washed and dried. The dried fiber may then be subjected to the various reactions given in the table, pp. 652–659, for recognition of the dye, this method of identifying colors by means of reactions on the dyed fiber being one of the most convenient.

A few of the vegetable colors as well as cochineal will dye wool directly, and these may be identified by reactions given in the table with the coal-tar dyes. Other vegetable colors, and the natural colors of fruits nearly always give a slight dull coloration or stain to the wool, but this is not, as a rule, to be mistaken for the vivid hues of the coal-tar dyes. Moreover most of the vegetable colors on the fiber turn green when treated with ammonia. Care should be taken to thoroughly wash the wool after the dyeing, so that colored particles simply held thereon mechanically may be removed.

Sostegni and Carpentieri † recommend a method of double dyeing, applicable when acid dyes are employed. The method consists in first boiling the wool in a dilute acid solution of the food sample as above described, after which the fiber is removed and boiled, first in very dilute hydrochloric acid solution, and then in water, till free from acid. The color is then dissolved from the fiber by boiling the latter in a weak ammoniacal solution, some of the colors being more readily dissolved than others. The fiber is then removed from the solution, the latter is acidified with hydrochloric acid, and the color fixed on a fresh piece of wool by boiling therein. The second dyeing fixes only coal-tar colors on the fiber, so that there is no fear of mistaking vegetable

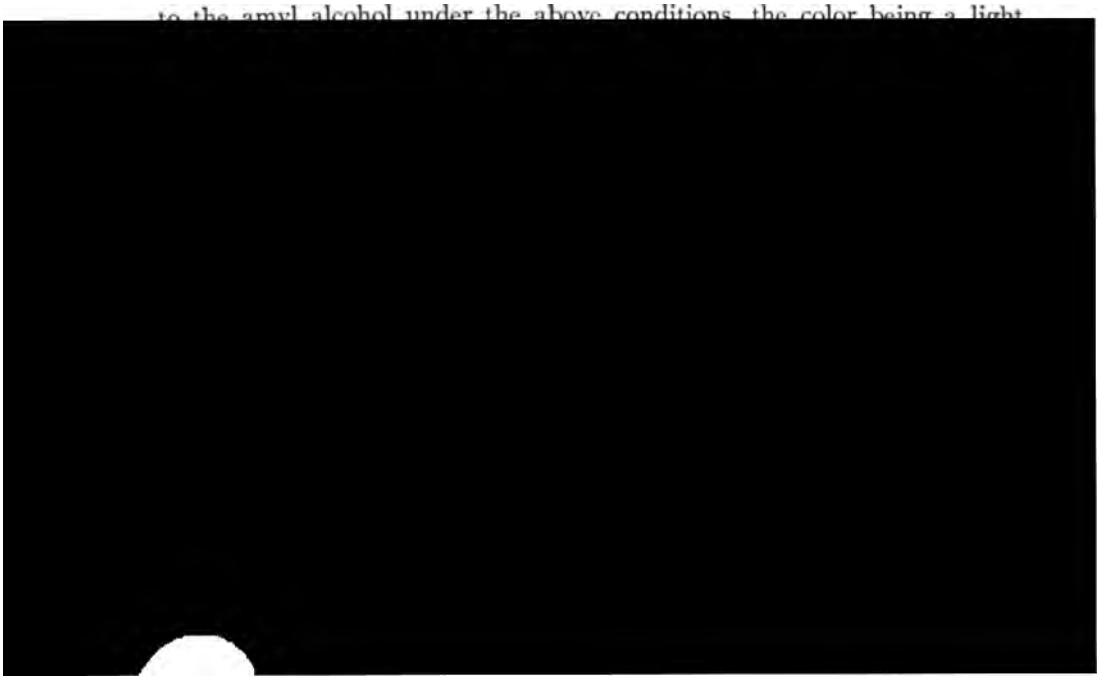
* Ztsch. anal. Chem., 28 (1889), 639.
† *Ibid.*, 35 (1896), 397.

colors therefor. Any color left on the first fiber, after treatment with ammonia, is probably due to the natural vegetable color of the sample, and is usually no more than a dull stain.

Vegetable Colors on Wool.—In case no color is directly fixed on the fiber by boiling wool in a solution of the sample, either neutral or acid, absence of coal-tar colors may be assumed. In this case it is sometimes advisable to boil strips of previously mordanted white cotton in an acid solution of the sample, to remove certain vegetable colors for purposes of testing on the fiber. The cotton is mordanted by boiling in a dilute (5%) solution of potassium bichromate.

Extraction of Colors from their Solution by Immiscible Solvents.—Methods based on this principle are in use in the municipal laboratory at Paris.* Sanglé-Ferrière uses the following method: 50 cc. of the wine or solution to be tested for color are rendered slightly alkaline by ammonia, and cautiously shaken with about 15 cc. of amyl alcohol. If acid dyes are present, they will be dissolved, and will impart to the amyl alcohol a distinct color.† Basic dyes also are dissolved, but when they are present the amyl alcohol solution is colorless. Remove the amyl alcohol by means of a separatory funnel, wash with water, and finally, if the alcohol is colored, dilute with about an equal volume of distilled water and evaporate on the water-bath with a piece of white wool. The wool should be kept in the solution till the odor of the amyl alcohol has disappeared, and, if not then colored, for a short time longer, as with some colors the wool will dye more readily in the aqueous solution than in the amyl alcohol. Remove the wool, and evaporate the solution to dryness. Test for color in the dried residue, and on the fiber also.

Orchil, like the acid colors, is extracted by, and imparts a coloration to the amyl alcohol under the above conditions, the color being a light



Fuchsin is indicated by a yellow-brown color with sulphuric acid, which by dilution with water becomes rose; *safranin*, by a green color becoming first blue, then red, when diluted with water, and *magdala red* by a dark blue color, turning red on the addition of water.

Basic colors are also extracted readily, according to Robin, by making the solution to be tested alkaline with sodium hydroxide, and shaking with acetic ether. The solvent is removed, washed, and evaporated with wool (on which the tests are to be made), or the evaporation is carried to dryness and the tests made on the residue.

Many coal-tar colors are extracted by amyl alcohol in acid solution, but some of the natural fruit colors are also dissolved under these conditions. The coal-tar dyes thus dissolved will, however, dye wool and the fruit colors will not. Fruit colors are not extracted from acid or alkaline solution by ether, nor from alkaline solution by amyl alcohol.

Robin's method for ascertaining whether acid colors are present consists in adding to the liquid to be tested an excess of calcined magnesia, and a little 20% mercuric acetate solution, the mixture being boiled and filtered. If the filtrate is colored, or if by the addition of acetic acid to the colorless filtrate a color is developed, a coal-tar dye is indicated.

Separation of Acid and Basic Colors with Ether.*—Acid and basic colors may be separated from their dilute aqueous solution, according to Rota, by means of ether as follows: To 100 cc. of the solution add 1 cc. of 20% potassium hydroxide and shake in a separatory funnel with several portions of ether. Basic dyes are dissolved by the ether, leaving behind as a rule the acid colors.† Wash the ether extract with faintly alkaline water, and shake out with 5% acetic acid. Some colors remain in the ether, others are dissolved in the acid. Separate the two solvents, and evaporate each to dryness on the water-bath.

The acid colors left in the slightly alkaline, aqueous solution after removal of the basic colors by ether as above, may, if desired, be separated into several groups by successive extraction, as follows: first slightly acidulate with acetic acid and extract with ether, then acidify with hydrochloric acid and again extract, and finally examine the residual solution for colors that are insoluble in ether. Thus erythrosin and eosin are soluble in ether when shaken with their aqueous solution made acid with hydrochloric acid, while acid fuchsin is insoluble.

---

* Analyst, 24, p. 45.

† A few acid dyes are exceptional in being soluble in ether with alkali, as for example, quinolin yellow and the sudans.

Separation of Colors from Dried Food Residues by Solvents.—This method is rarely employed, excepting in the case of colors insoluble in water, but soluble in ether or alcohol. The dried pulp of canned vegetables, ketchups, etc., may be acidified with hydrochloric acid, and the color extracted therefrom directly with alcohol. In this case however, there is no obvious advantage over the previous methods of dyeing the fiber directly in the acid solution of the sample.

Girard's Tests for Acid Fuchsin.* — Add 2 cc. of 5% potassium hydroxide to 10 cc. of the wine or other solution to be tested, or enough of the alkali to neutralize the acid. Then add 4 cc. of 10% acetate of mercury and filter. The filtrate should be alkaline and colorless. If the solution remains uncolored after acidifying with dilute sulphuric acid, no acid fuchsin is present. If, however, there is produced a red to violet coloration, and no other coal-tar colors have been found by the amyl alcohol extraction, the presence of acid fuchsin is shown.

Bellier's Test for Acid Fuchsin.—Presence of acid fuchsin is indicated by adding to 20 cc. of wine or other solution to be tested about 4 grams of freshly precipitated yellow oxide of mercury, boiling and filtering. The filtrate, if acid fuchsin is present, is colored red, tinged with violet.

According to Blarez, all red coal-tar colors, with the exception of acid fuchsin, and all red vegetable colors are completely decolorized by acidulating their aqueous solution with tartaric acid, and digesting with dioxide of lead.†

CLASSIFICATION AND IDENTIFICATION OF COAL-TAR DYES.

Various excellent schemes have been prepared for identifying unknown colors by their characteristic reactions, first grouping them into classes

the dyes. The colors are divided into two main groups, according to whether or not they are reducible by stannous chloride. These two groups are each further subdivided into two classes, the reducible colors being classed according to whether the color remains unchanged, or is restored by treatment with ferric chloride, and the non-reducible colors according to their action with potassium hydroxide.

The tests are carried out on a dilute aqueous or alcoholic solution of the coloring matter, the strength being about 1 in 10,000. Treat about 5 cc. of this solution with 4 or 5 drops of concentrated hydrochloric acid and about as much stannous chloride in a test-tube, shake the mixture, and heat if necessary to boiling. With some colors the process of decolorization is a slow one, especially if the solution is too concentrated, and it is well to repeat the experiment, if in doubt, diluting the original sample still further with water. Tin in solution in concentrated hydrochloric acid may be employed instead of stannous chloride, if desired.

Here, as in all cases of color testing, it is well to make comparative tests with known colors.

CLASSIFICATION OF ORGANIC COLORING MATTERS.

[A portion of the aqueous or alcoholic solution is treated with HCl and $SnCl_2$.]

Complete decolorization. Reducible coloring matters. Colorless solution is treated with Fe_2Cl_6, or shaken with exposure to air.		**The color changed no further than with HCl alone. Nonreducible colors. A part of original solution is mixed with 20% KOH and warmed.**	
The liquid remains unchanged. Coloring matters not reoxidizable.	The original color restored. Reoxidizable coloring matters.	Decolorization, or a precipitate. Imidocarbo-quinone coloring matters.	No precipitation. Liquid becomes more colored. Oxycarbo-quinone coloring matters.
CLASS I.	CLASS II.	CLASS III.	CLASS IV.
Nitro nitroso, and azo colors, including oxyazo and hydrazo colors. Picric acid, naphthol yellow, ponceau, Bordeaux, and Congo red.	Indogenide and imidoquinone coloring matters, methylene blue, safranin, indigo carmine.	Amido-derivatives of di and triphenyl methane, auramins, acridins, quinolins, and color derivatives of thio benzenil. Fuchsin, rosanilin, auramin.	Nonamide diphenyl methane, oxy-ketone, and most of natural organic coloring matters. Eosins, aurin, alizarin.

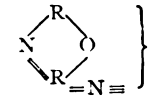
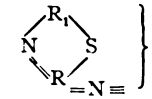
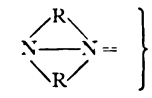
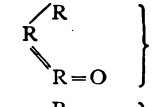
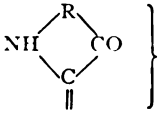
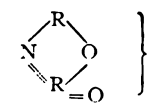
RACTERISTICS OF ORGANIC COLORING MATTERS.

ASS I.—REDUCED BY $HCl+SnCl_2$ AND NOT REOXIDIZABLE.

				Types.
nines; soluble in ether in the presence of KOH.			$-N=R=N\langle^{O.}_{OH.}$	e.g., Aurantia.
-phenols; in ether resence of	Nonsulphonated; soluble in ether in presence of acetic acid.		$O=R=N\langle^{O.}_{H.}$	Victoria yellow.
	Sulphonated; insoluble in ether.			Naphthol yellow.
lphonated; insoluble in water; soluble in alcohol· soluble in ether in of acetic acid.				Dioxin (L).
onated; soluble in water; insoluble in ether.				Naphthol green.
ed; shaken with dilute acetic acid it the original color. Basic col- atters.	Nonsulphonated amido-azo coloring matters.		$-N=R=N-NHR_1$.	Bismarck brown.
ed solution; not yielding its color acetic acid. Neutral coloring	Oxyazo coloring matter, without carboxyl.		$O=R=N-HR_1$.	Sudan I (A).
less soluelds nothcetic acid. oring mat-	Non sulphonated; extracted by ether from dilute solution in acetic acid.	Oxyazo coloring matters, with carboxyl group.	Indirect for cotton wool.	Diamond yellow (By)
			Direct for cotton wool.	Chrysamin.
	Sulphonated; not extracted by ether from solution in dilute acetic acid.	Nonamido compounds; unaltered by HNO_2.	Indirect for cotton wool.	Bordeaux B (A).
			Direct for cotton wool.	Azo blue (A).
		Amido compounds; changed by HNO_2.	Indirect for cotton wool.	Solid yellow N (P).
			Direct for cotton wool.	Congo red (A).

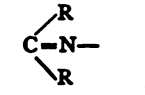
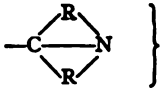
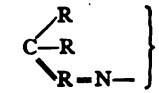
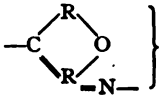
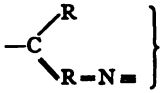
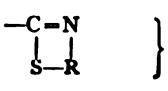
CLASS II.—REDUCED BY $HCl + SnCl_2$ AND REOXIDIZABLE.

The aqueous or alcoholic solution is treated with KOH and extracted with ether. The ethereal solution washed with water has the annexed characteristics.

				Types.
The ethereal solution is colored or colorless, and yields the original color to 5% acetic acid. Basic coloring matters fixed on wool in alkaline bath.	The solution is readily reduced by $HCl + SnCl_2$ in the cold.	Oxyazines (no sulphur).		Nile blue A (B).
		Thiazins (sulphur).		Methylene blue.
	The colored solution is reduced but slowly and incompletely, even on warming, and with the addition of much $SnCl_2 + HCl$.	Indulins; blue color with concentrated H_2SO_4. Blue on dilution.		Indulin soluble in alcohol.
		Safranins; green color with H_2SO_4. On dilution blue, then violet.		Safranin T extra (A).
Colored; does not yield the color to acetic acid. Neutral coloring matters. Insoluble in water. Soluble in alcohol. Fixed on fibers in bath.	Blue coloring matters changed by HCl on warming.	Indophenols.		Indophenol.
	Red or blue coloring matters. Unaltered by HCl. With HNO_3 yield isatin.	Indogenides.		Indigotin.
Uncolored; yields nothing to acetic acid. Acid coloring matters. Soluble in water. Fixed on wool in acid bath.	Nonsulphonated. Soluble in ether in presence of acetic acid.	Oxazones.		Fluorescent blue; orcein.
	Sulphonated. Insoluble in ether under all circumstances.	Reduced by $SnCl_2 + HCl$.	Sulphonated indogenides.	Indigo carmine.
			Sulphonated thiazins.	Thiocarmine R (C).
		Not reduced by $SnCl_2 + HCl$.	Sulphonated indulins.	Soluble nigrosin.

RISTICS OF ORGANIC COLORING MATTERS—(*Continued*).

DUCED BY SnC_2+HCl. CONTAINING THE IMIDO-QUINONE CARBON CHROMOPHORE —N=R=C=.

		Types.	
nonfluorescent ethereal solution. yielded to acetic acid nonfluores- queous solution is decolorized by composed by HCl.	Auramins.		E.g. Auramin O (B).
ethereal solution. Green fluores- ous solution precipitated by KOH, d by HCl. Turns red with HNO_3.	Acridins.		Phosphin.
or colored ethereal solution. Non- Color yielded to acetic acid— t, blue, and green without fluo- queous solution usually decolor- ing with KOH, and colored yel- (excepting fuchsin).	Fuchsins (nonsulphonated).		Fuchsin.
olution colorless and nonfluores- acid colored rose and fluoresces.	Pyronins (colored yellow by HCl. Direct for cotton wool).		Pyronin (G).
olution decolorized with KOH.	Rhodamins (non-sulphonated. Unaltered by HCl).		Rhodamin S (By).
The ethereal solution is yellow, nd nonfluorescent. Alcoholic so- tion yellow, nonfluorescent, and naltered by aqueous acids and al- alies.	Quinone-phthalones.		Quinolin yellow A (soluble in alcohol).
Yellow coloring matters. No fluorescence in water. Unaltered by aqueous acids and alkalies.	Sulphonated quinone-phthalones.		Quinolin yellow A (soluble in water).
Reddish violet, blue, or green coloring matters. Usually decolorized by KOH, little changed by HCl.	Sulphonated fuchsins.		Fuchsin S (B).
Red or violet coloring matters. Soluble in water with fluorescence. Precipitated by HCl. Changed but little, or not at all, by KOH.	Sulphonated rhodamins.		Violamin R (M).
Brownish yellow or orange coloring matters. Aqueous solution ± fluorescent. Fixed directly on silk, wool, and cotton.	Thiazoles.		Primulin (B).

CLASS IV.—COLORING MATTERS NOT REDUCED BY $SnCl_2+HCl$. CONTAINING THE OXY-QUINONE CARBON CHROMOPHORE O=R=C=.

The alcoholic solution of the coloring matter treated with a few drops of a dilute solution of Fe_2Cl_6 (1:1000).

							Types.
Remains unaltered. Nonamido triphenylmethane coloring matters. Usually soluble in water and direct for wool.	The coloring matter is dissolved or suspended in boiling water.		Not directly fixed on wool. Most of them insoluble in water. Soluble in alcohol without fluorescence.	Aurins.		Aurin.
			Fixed directly on wool. Most of them soluble in water and alcohol. Fluorescence.	Phthaleins.		Eosin.
Changes to green or olive green. Oxy-ketone coloringmatters. Most of them insoluble in water and indirect for fibers.	The original coloring matter is treated with faintly alkaline water (KOH, 1 per cent).	Dissolves with yellow or reddishyellowcolor. Monoketones.	The alkaline solution treated with excess of HCl.	Inclined to decolorization, especially on warming (with decomposition).	Benzophenones.		Alizarin yellow A (B).
				Colored intense yellow without decomposition.	Flavones.		Quercetin.
		Dissolves with red, reddish violet, green, or blue color. Diketones (quinones).	The alkaline solution acidified with acetic acid.	The free coloring matter precipitated. Usually soluble in ether, and indirect for fibers.	Nonsulphonated anthraquinones.		Alizarin.
				Coloring matters remain in solution. Insoluble in ether, fixed directly on wool.	Sulphonated anthraquinones.		Sulphonated alizarin (alizarin red).

Direct Identification of Colors.—In identifying the colors commonly used in food, it is rarely necessary to carry out such involved processes of analyses as are rendered necessary by Rota's scheme. It is frequently possible to ascertain the color or group of colors present by making direct tests with various reagents, either on the dyed fiber, or on the dry coloring matter, or in a solution containing it.

Many tables for this purpose are prepared, but they are never complete by reason of the many new dyestuffs constantly introduced. Such tables are to be found in Allen's "Commercial Organic Analysis," and in Schultz and Julius's "Systematic Survey of the Coal Tar Colors." While it is true that the limitation of the dyes suitable for purposes of food coloring imposes a somewhat lighter task on the food analyst than that of the chemist who has to deal with all varieties of commercial colors, yet it is obviously impossible to make a complete list covering even the restricted field of food colors. Doubtless there are colors long well known that would serve admirably for this purpose, but have never yet been tried.

Mainly from such sources as the above comprehensive tables of colors and their reactions, the writer has compiled the table on pp. 652–659, taking as a basis the scheme of Allen.* This table includes about fifty selected coloring matters, which are adapted for, and have been found in, foods by various analysts, as listed in state and government reports, as well as in laws of various countries dealing with food colors. This table will at least contain the colors most commonly met with, and will nearly always serve, if not to identify the exact dye, to aid in classifying it. In case the analyst wishes to identify the color, he should be provided, for standards, with as complete a collection of known purity dyestuffs as possible covering the colors he is likely to

Reagents.—In applying tests on the fiber, the reagents commonly used are as follows: Concentrated hydrochloric acid, concentrated sulphuric acid, sodium hydroxide (10% solution), strong ammonia (28%), a hydrochloric acid solution of stannous chloride, and concentrated nitric acid. The tests should be made on pieces of the fiber in small porcelain evaporating-dishes, which more readily than test-tubes show exact shades of color. In cases of suspected fluorescence, test-tubes should be used. Nitric acid is conveniently applied by a glass rod to the fiber. The stannous chloride should first be allowed to act in the cold. If no change occurs, gentle heat should then be applied, and finally boiling.

DENTIFICATION OF COLORS ON THE FIBER.

Character of Dyestuff.		Acid or Basic.	Hydrochloric Acid.		Sulphuric Acid.	
Dry.	Solution.		Fiber.	Solution.	Fiber.	Solution.
tle-green rystals	Red	Basic	Yellow *	Yellow	Brown * yellow	Yellow
n glisten- powder	Crimson	Acid	Paler	Pink	Paler	Pink
vn powder	Red	Basic	Blue	Yellow	Green	Green
uish-red rystals	Red	Acid	Yellow	.....	Yellow	Yellow
d-brown owder	Crimson	Acid	Dark-red violet *	Lilac	Violet-black	Violet-black
wn-yellow owder	Red, greenish fluorescence	.....	Yellow	.....	Yellow	
vn powder	Red	.....	Orange	.....	Yellow	Orange
lish brown	Red	Acid	Violet †	Colorless ‡	Blue	Blue
vn powder	Orange	Acid	Violet	Colorless ‡	Green	Green
vn powder	Orange	Acid	Violet	Colorless ‡	Green	Green
vn powder	Brown	Acid	Black	Colorless	Black	Blue
l powder	Red	Acid	Dark-red violet	Lilac	Dark-red violet	Lilac
vn powder	Red	Acid	Decolorized	.....	Brown	
vn powder	Red	Acid	Blue	Blue	Blue	Blue
arlet-red owder	.....	Acid	Darker	.....	Crimson	
.....	Reddish brown	Acid	Purple	.....	Violet	Violet
vn powder	Purple	Acid	Crimson	Pink	Violet	Purple
vn powder	Magenta-red	Acid	Darker	Pink	Violet	Violet
lish-brown owder	Magenta	Acid	Darker	Pink	Violet	Violet

Fuchsin is indicated by a yellow-brown color with sulphuric acid, which by dilution with water becomes rose; ***safranin***, by a green color becoming first blue, then red, when diluted with water, and ***magdala red*** by a dark blue color, turning red on the addition of water.

Basic colors are also extracted readily, according to Robin, by making the solution to be tested alkaline with sodium hydroxide, and shaking with acetic ether. The solvent is removed, washed, and evaporated with wool (on which the tests are to be made), or the evaporation is carried to dryness and the tests made on the residue.

Many coal-tar colors are extracted by amyl alcohol in acid solution, but some of the natural fruit colors are also dissolved under these conditions. The coal-tar dyes thus dissolved will, however, dye wool and the fruit colors will not. Fruit colors are not extracted from acid or alkaline solution by ether, nor from alkaline solution by amyl alcohol.

Robin's method for ascertaining whether acid colors are present consists in adding to the liquid to be tested an excess of calcined magnesia, and a little 20% mercuric acetate solution, the mixture being boiled and filtered. If the filtrate is colored, or if by the addition of acetic acid to the colorless filtrate a color is developed, a coal-tar dye is indicated.

Separation of Acid and Basic Colors with Ether.*—Acid and basic colors may be separated from their dilute aqueous solution, according to Rota, by means of ether as follows: To 100 cc. of the solution add 1 cc. of 20% potassium hydroxide and shake in a separatory funnel with several portions of ether. Basic dyes are dissolved by the ether, leaving behind as a rule the acid colors.† Wash the ether extract with faintly alkaline water, and shake out with 5% acetic acid. Some colors remain in the ether, others are dissolved in the acid. Separate the two solvents, and evaporate each to dryness on the water-bath.

The acid colors left in the slightly alkaline, aqueous solution after removal of the basic colors by ether as above, may, if desired, be separated into several groups by successive extraction, as follows: first slightly acidulate with acetic acid and extract with ether, then acidify with hydrochloric acid and again extract, and finally examine the residual solution for colors that are insoluble in ether. Thus erythrosin and eosin are soluble in ether when shaken with their aqueous solution made acid with hydrochloric acid, while acid fuchsin is insoluble.

---

* Analyst, 24, p. 45.

† A few acid dyes are exceptional in being soluble in ether with alkali, as for example, quinolin yellow and the sudans.

TIFICATION OF COLORS ON THE FIBER—*Continued.*

Character of Dyestuff.		Acid or Basic.	Hydrochloric Acid.		Sulphuric Acid.	
Dry.	Solution.		Fiber.	Solution.	Fiber.	Solution.
own paste	Red-brown	Acid	Crimson	Crimson	Crimson	Crimson
wn powder	Purple	Acid	Crimson	Pink	Violet	Violet
k-red paste	Red		No change	Red	Purple	Purple
k-red wood	Red		Dark red	Pink	Brown	Yellow
k-red wood	Red		Redder	.....	Red-brown	Dirty brown
ry-red powder	Yellow-red		Decolorized	.....	Decolorized	
ry pinkish insect	Deep red		Orange	Orange	Pink	Pink
.....	.....	.....	Lighter	Red	Dirty brown	
low crystals	Yellow	Acid	Decolorized	Yellow	Decolorized	
nge powder	Orange	Acid	Decolorized			
nge-yellow plates	Yellow	Acid	Decolorized	Colorless	Decolorized	
nge-yellow powder	Yellow	Acid	Decolorized	Colorless	Decolorized	
ed-brown powder	Orange-brown	Basic	Red	.....	.....	Yellow
low powder	Yellow	Acid	Red	.....	Brown	
ellow-red powder	Orange	Acid	Violet	Violet	Violet	Violet
nge-yellow powder	Orange	Acid	Red	Pink	Violet	Violet

	Sodium Hydroxide.		Ammonia.		Spot with Nitric Acid.	Stannous Chloride.	Alcohol.
	Fiber.	Solution.	Fiber.	Solution.			
RED COLORS (*Continued*).							
Orchil substitute V.	Maroon	.....	Maroon	.....	Yellow	Lighter	
Naphthion red.							
Scarlet 6R.	Brown	.....	.....	Pink	Yellow		
Orchil *	Purple	.....	Purple	.....	.....	Decolorized	Purple
Archil orseille.							
Brazil wood †	Maroon	Violet	Violet	Violet	Yellow	Violet	Pale yellow
Barwood.	Purple	Colorless	Purple	Colorless	Olive	No change	Red
Safflower ‡	Pale yellow	.....	Pink	.....	.....	Yellow	No action
Cochineal §	.....	Purple	.....	Purple	Yellow	Orange	No action
Cudbear *	Violet	Violet	Violet	Violet	Yellow, red rim	Decolorized	Bluish red
YELLOW AND ORANGE COLORS.							
Picric acid.	Orange	Yellow	Paler	Yellow	.....	Decolorized	Yellow
Victoria yellow ‖	.....	.....	No change				
Anilin orange.							
Dinitrocresol.							
Naphthol yellow ¶	Orange	Yellow	Paler	Yellow	.....	Decolorized	Yellow
Martius yellow.							
Naphthol yellow S.	Paler	Yellow	No change	Yellow	.....	Bleached	No action
Acid yellow.							
Chrysoidine yellow.....	Paler	.....	Yellower	.....	.....	Decolorized	Yellow
Fast yellow.	No action	.....	No action	.....	Red	Decolorized	Pale yellow
Acid yellow.							
Orange II.	Deep red	.....	No action	.....	.....	Decolorized	No action
Tropæolin OOO No. 2.							
Orange III.	Yellowish	.....	No change	Yellow	.....	Decolorized	Pale yellow
Methyl orange.							
Tropæolin D.							

* Dyes wool in neutral or slightly acid solution.
† Does not dye wool unless mordanted with alum or chrome.
‡ Does not dye wool, but dyes cotton in alkaline solution.
§ Dyes wool by tin mordant.
‖ Warm water extracts color.
¶ KCN—red.

TIFICATION OF COLORS ON THE FIBER—*Continued*

Character of Dyestuff.		Acid or Basic.	Hydrochloric Acid.		Sulphuric Acid.	
Dry.	Solution.		Fiber.	Solution.	Fiber.	Solution.
.....	.....	Acid	Violet	Violet	Violet	Violet
ull-yellow powder	Pale yellow	Acid	Brown	Brown	Brown	Dark red
iery-red powder	Orange-yellow	Acid	Red	Red	Orange-yellow	Red
rown-red es or rolls	Cream	.....	No change	Red	Blue	Blue
rk-yellow powder	Yellow	.....	Brown	Colorless	Brown	Brown
low heavy powder	Yellow	.....	Decolorized	Yellow	Duller	
ow or buff powder	Yellow	.....	No change	Yellow	Yellow	Yellow
low Brazil wood	.....	.....	Orange	Orange	Brown	Brown
all bits of od. Fustet	Yellow	.....	No change	Yellow	Brown	Brown
.....	Pale yellow	.....	No change	Yellow	Bright yellow	
llow-green y berries	Yellow	.....	No change	Yellow	Brown	Yellow
wnish-yel- w powder	Orange	Acid	Purple	Purple	Dark purple	
lowish-red powder	Reddish yellow	Acid	Scarlet	Pink	Crimson	Red
wn powder	Yellow	Acid	Violet	.....	Violet	Violet
wn powder	Brown	Acid	Darker	Yellow	Purple	Purple
wn powder	Brown	Basic	Reddish	Red	Reddish	Red
.....	Brown	.....	Decolorized	.....	No action	

	Sodium Hydroxide.		Ammonia.		Spot with Nitric Acid.	Stannous Chloride.	Alcohol.
	Fiber.	Solution.	Fiber.	Solution.			
YELLOW AND ORANGE COLORS.							
Orange IV.	No action	.....	No change	Yellow	.....	Decolorized	Yellow
Tropæolin OO.							
Primulin orange.	Dark brown	.....	No action	.....	Brown	Decolorized	Pale orange
Crocein orange.	Browner	.....	Slightly darker	.....	.....	Decolorized	
Ponceau 4GB.							
Annatto *	No action	.....	No action	.....	.....	Decolorized	Yellow
Turmeric.	Brown	Brown	Brown	Orange	Yellow	Brown	Yellow
Chrome yellow †	Redder	Yellow	No action	.....	.....	Decolorized	No action
Quercitron bark ‡	No change	Yellow	No change	Yellow	Blue	No change	No action
Old fustic ‡	No change	.....	No change	Yellow	Pale yellow	Orange	No action
Young fustic ‡	Brown	.....	Brown	.....	Dark brown	No action	No action
Weld ‡	No change	Yellow	No action	.....	No action	No change	No action
Persian berries ‡	No change	Yellow	.....	Pale yellow	Brown	Brown	No action
Metanil yellow.	Brighter	.....	No change	.....	Red	Brown	Pale yellow
Tropæolin.							
Victoria yellow.							
Orange G.	Terra cotta	.....	No change	.....	Yellow	Decolorized	
Chrysamine R	Darker	.....	Darker	Pink	Red	.....	Yellow
BROWN COLORS.							
Acid brown G.	Darker	.....	Brighter	Brown	Yellow	No change	Brown
Bismarck brown.	Yellower	Colorless	No change	Brown	.....	Decolorized	Pink
Vesuvine.							
Manganese brown §	No action	.....	No action	.....	.....	Decolorized	No action

* $K_3Fe(CN)_6$—blue.
† H_2S—black.
‡ Fe_2Cl_6—olive.
§ Ash contains Mn.

TIFICATION OF COLORS ON THE FIBER—*Continued.*

Character of Dyestuff.		Acid or Basic.	Hydrochloric Acid.		Sulphuric Acid.	
Dry.	Solution.		Fiber.	Solution.	Fiber.	Solution.
en metallic powder	Violet	Basic	Brown	Amber	Brown	Amber
ck powder	Violet	.....	Black	Colorless	Black	Blue
nze powder	Violet	.....	Violet	Blue	.....	Dark blue
.....	Indigo	.....	No change	.....	.....	Blue
eep-blue powder	Blue	.....	No action	.....	No action	
.....	.....	.....	Decolorized	.....	Decolorized	
nze powder	Blue	.....	Decolorized	Blue-green	Green	Green
ay-brown powder	Green	.....	Yellow	Red	Brown	Brown
en crystals	Bluish green	Basic	Orange	Orange	Bleached	Orange
ark-green powder	Green	.....	Yellow	Yellow	Olive	Drab

	Sodium Hydroxide.		Ammonia.		Spot with Nitric Acid.	Stannous Chloride.	Alcohol.
	Fiber.	Solution.	Fiber.	Solution.			
VIOLET-BLUE AND GREEN COLORS.							
Methyl violet	Decolorized	Colorless	Lilac	.....	.....	Green	Violet
Azo blue.	Red	Pink	Dark violet	Red	.....	Decolorized	No action
Indulines soluble.	.....	Violet decolorized by zinc dust	.....	AsNaOH	Dark green	Violet solution	Blue-violet
Fast blue R.							
Indigo.	No change	.....	No change	.....	Yellow	Paler, solution yellow	Blue
Prussian blue *	Brown	.....	No action	.....	Green	No action	No action
Ultramarine blue †	No action	.....	No action	.....	.....	Decolorized	No action
Methylene blue ‡	Violet	.....	No action	.....	Green	Decolorized	Greenish blue
Resorcin green *	Darker	Greenish	No action	.....	Brown	Paler	No action
Malachite green	Decolorized	.....	Decolorized	.....	.....	Decolorized	Green
New Victoria green.							
New green.							
Naphthol green	Brighter	Olive	No action	.....	Orange	Decolorized	Pale blue

* Ash contains Fe. † Blue ash. ‡ $CaOCl_2$—decolorized.

ABBREVIATIONS OF FIRMS.

Ber. —Berlin Aniline Co.
D.H.—Durand, Huguenin & Co. Basle.
C. —L. Cassella & Co., Frankfort.
M. —Meister, Lucius & Brunig, Höchst.
By. —The Bayer Co., Elberfeld.
B.S.S.—Brooke, Simpson & Spiller, London.
P. —A. Poirier et G. Dalsace, Paris.
O. —K. Oehler, Offenbach.
H. —Read, Holliday & Sons, Huddersfield.
Sch. —The Schoellkopf Aniline and Chemical Co., Buffalo.

REFERENCES ON COLORS.

ARATA, P. N. (Specielle analytische Methoden.) Zeits. anal. Chem., 28, 639.

BELLIER, J. Detection of Artificial Coloring Matters in Wine. Ann. de Chim. Anal., 5, 1900, 407; Abs. Analyst, 26, 1901, 42.

BENEDIKT, R., and KNECHT, E. The Chemistry of the Coal-tar Colors. London, 1889.

DOMMERGUE, G. Detection of Colors on Dyed Wool. Monit. Scient., 33, 25; Abs. Jour. Soc. Chem. Ind., 8, 216.

FOL, F. Testing of Dyestuffs. Jour. Chem. Soc., 28, 1875, 193.

GIRARD, P. Couleurs Employés dans les Matières Alimentaires. Analyse des Matières Alimentaires (Girard et Dupré), p. 689. Paris, 1894.

GREEN, A. G. On the Qualitative Analysis of Coal Tar Coloring Matters. Jour. Soc. Chem. Ind., 12, 1893, p. 3.

LEEDS, A. R. Tabellarische Uebersicht der künstlichen organischen Farbstoffe. Berlin, 1894.

MARTINON, B. Jour. Soc. Dyers, 3, 124.

NIETZKI, R. Chemie der organischen Farbstoffe. Berlin, 1901.

POSETTO, G. Composition of Vegetable Coloring Matters for Use in Confectionery. Zeits. Nahr. Unters. u. Hygiene, 9, 1895, 150.

RAWSON, C., KNECHT, E., and LOWENTHAL, R. A Manual of Dyeing. London, 1893.

RAWSON, GARDNER, and LAYCOCK. A Dictionary of Dyes, Mordants, etc. 1890.

REICHELMANN and LEUSCHER. Detection of Coal Tar Colors in Pastry, Cakes, Fruit Products, etc. Zeit. für öffentl. Chem., 8, 1902, 204; Abs. Analyst, 27, 1902, 276.

ROTA, A. R. A Method of Analyzing Natural and Artificial Organic Coloring Matters. Analyst, 24 (1899), 41. From Chem. Zeit., 1898, 437.

SCHULTZ, G., u. JULIUS, P. Taballarische Uebersicht der künstlichen organischen Farbstoffe. 1897.

—— Translated by Green, A. G. A Systematic Survey of the Organic Coloring Matters. 1894.

SPAETH, E. Foreign Coloring Matters in Fruit Juices. Zeits. für Unters. der Nahr. u. Genuss., 2, 1899, 633.

SOSTEGNI, L., and CARPENTIERI, F. (Specielle analytische Methoden.) Zeits. anal. Chem., 35 (1896), 397.

SPILLER, J. Identification of the Coal Tar Colors. Analyst, 6, 1881, 22.

CHAPTER XVII.

FOOD PRESERVATIVES.

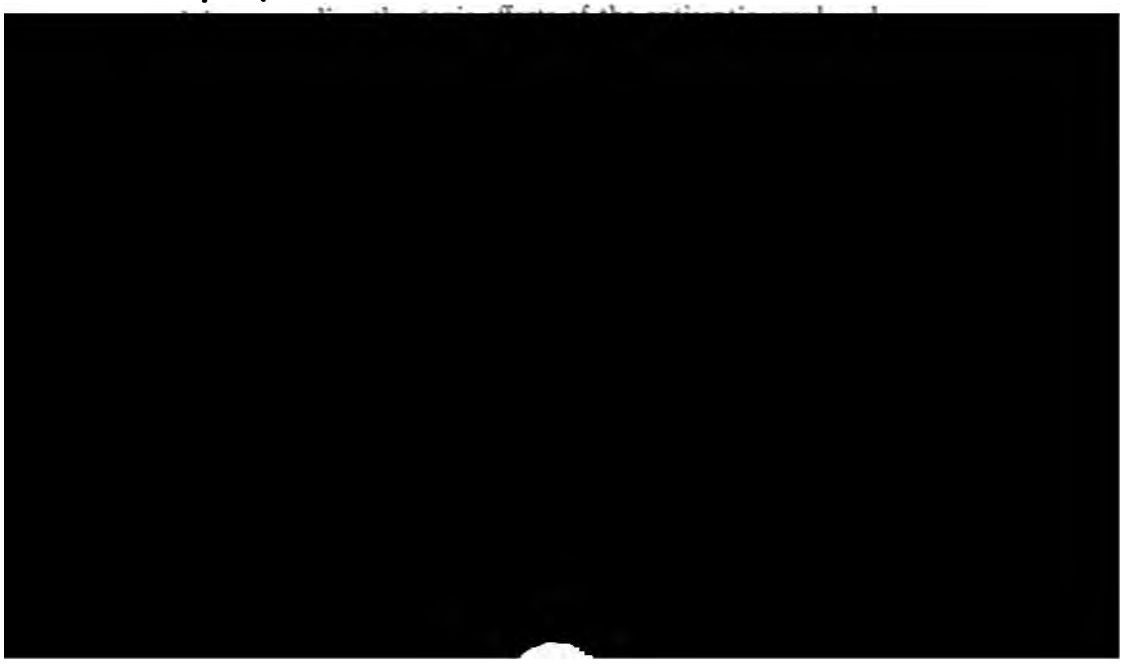
Preservation of Food.—Various processes have from ancient times been known and used for arresting the fermentative changes which food products in their natural state undergo on long standing. These processes include pickling with vinegar, drying, smoking, salting, preserving with sugar, and finally in the employment of heat in sterilizing and pasteurizing, and of low temperature as in cold storage. All of them are still in use, and are universally regarded as unobjectionable. In addition to these old and well-known methods of food preservation is the comparatively modern practice of arresting fermentation by the use of such antiseptic chemical agents as formaldehyde, beta-naphthol, boric, salicylic, benzoic, and sulphurous acids or salts of these acids, etc., in regard to the wholesomeness of which there is considerable difference of opinion. These substances depend for their efficiency on the more or less complete inhibition of bacterial growth. Nearly all exert a powerful antiseptic influence, to such an extent that to accomplish their object only small quantities need be used in food.

Apart from their toxic effects, a marked difference naturally exists between the employment of such substances as salt, sugar, and vinegar for food preservation, all of which are in themselves foods, and in the use of chemical agents that have no food value. The advocates of the use of chemical antiseptics claim that there are no authentic instances on record of injury from the use of such small quantities of these substances as are necessary to arrest decay, while there are many cases of injury arising from the use of foods which, while apparently wholesome, have undergone such fermentation as to develop ptomaines or other harmful toxins, and that because antiseptics prevent such spoiling of the food, their use is decidedly beneficial; that there is, besides, no more reason why a prejudice should exist against the employment of these

newer chemicals than against saltpeter, which has long been used in the corning of meat, or against the cresols and phenols left as a product of smoking.

The opponents to their use assert, that the addition to food of such antiseptic substances as prevent its decay also serves to retard the digestive processes when the food is eaten; that many of these substances are drugs, and as such cannot fail even in small quantities to exercise a toxic effect of some sort on the system; that finally their use is objectionable, as allowing the employment in certain foods of old materials that have in some cases already undergone incipient decomposition before the addition of the antiseptic, and are thus unwholesome.

Regulation of Antiseptics in Food.—In the absence of legislation directly prohibiting the use of any of the above-named antiseptics, and in view of the difference of opinion regarding their toxic effects when present in small quantities, it is difficult to maintain a complaint under the general food laws as they exist in most states, basing the complaint solely on their harmfulness. In some localities certain antiseptics are specifically allowed and others are prohibited. Some of the states, as, for example, Massachusetts, have special laws under which it is required that in the case of all foods thus treated, the name and percentage of such antiseptics as are used must appear plainly on labels of the packages or containers thereof, such a provision being based on the assumption that the general public should be informed of what they are buying, where any doubt exists as to the wholesomeness of any ingredient present. Where such laws as these are in force, the chemist's task is comparatively easy, in that conviction in court is not dependent on his individual



measures would seem to be along the line of compelling the honest labelling on the package of the preservatives employed.

Commercial Food Preservatives.—A large number of commercial preparations are sold for purposes of preserving specific articles of food and are put out under trade names that usually convey no suggestion of their true character. Some of these consist of a single antiseptic substance, such as salicylic acid, ammonium fluoride, calcium sulphate, borax, or benzoic acid, while others are mixtures of several antiseptics, of which the following are typical examples, showing their composition as found, together with the amount of the mixture to be employed.

A. For preserving sausage meat, using 8 ounces per 100 pounds of meat:

Borax	36%
Salt	46%
Saltpeter	18%

(Colored with an anilin dye.)

B. For preserving cider and ketchup.

A 34% solution of beta-naphthol in alcohol, using 2 fluid ounces to 45 gallons of cider, or 1½ ounces to 10 gallons of ketchup.

C. For preserving beer, using 1⅓ ounces per barrel of beer:

Salt	45%
Salicylic acid	27%
Sodium carbonate and salicylate	28%

D. For preserving chopped meats, using 1 ounce to 50 pounds of meat:

Sodium sulphite	65%
Borax	35%

E. Effective for curing beef, hams, tongues, bacon, pig's feet, etc.:

Borax	28%
Boric acid	12%
Sodium chloride	35%
Potassium nitrate	25%

F. For preserving milk and cream:

Boric acid	75%
Borax	25%

G. For preserving jellies, jams, preserves, mince-meat, and syrups, using from 1 to 2 ounces of preservative to 100 pounds of product:

Sodium benzoate	50%
Boric acid	40%
Sodium chloride	5%
Sodium bicarbonate	5%

H. For preserving ketchup and tomato pulp, using from 6 to 8 ounces to 45 gallons of the product:

Sodium benzoate	50%
Sodium chloride	40%
Sodium sulphite	10%

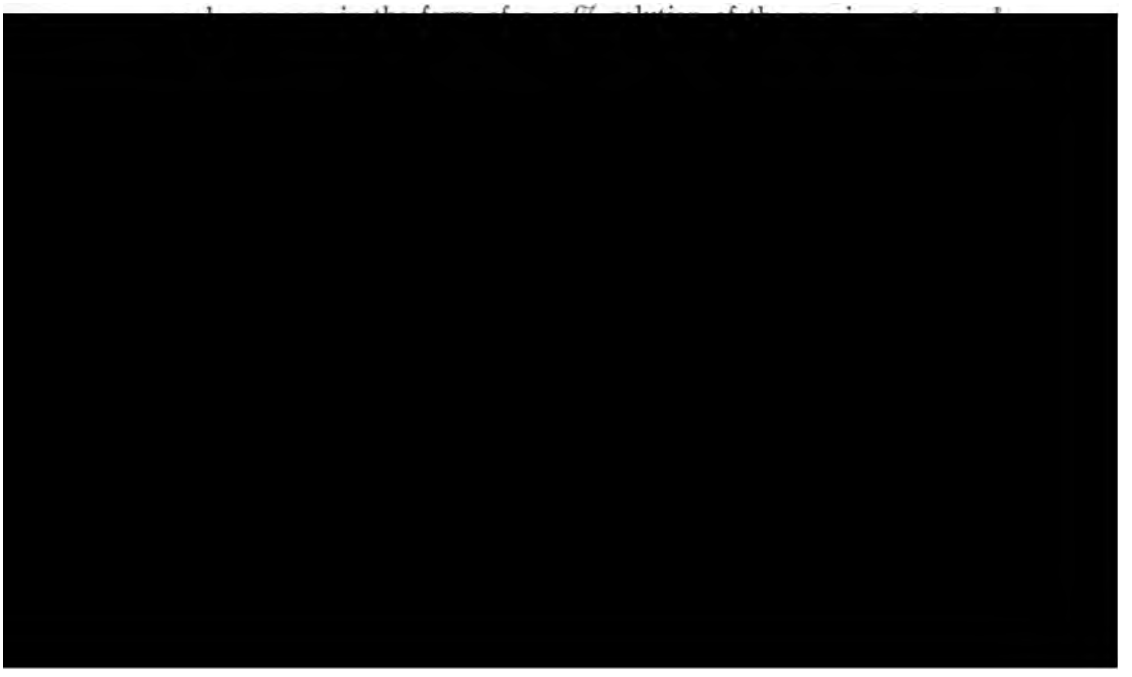
I. Effective for keeping butter from becoming tainted or rancid, also for salt codfish, using 8 to 12 ounces per 100 pounds butter:

Boric acid	25%
Borax	50%
Sodium chloride	25%

J. For preserving eggs (surface application). A saturated solution of salicylic acid in 3 quarts of water, 1 quart strong alcohol and 7 ounces of glycerin.

FORMALDEHYDE.

Formaldehyde (HCHO) is a gas formed by the action of a red-hot spiral of platinum wire on vaporized methyl alcohol. It is also produced by the dry distillation of calcium formate. In the market it com-



sweet for four days in summer weather, when under ordinary conditions, the milk untreated would curdle in less than forty-eight hours.

Determination of Formaldehyde in the Commercial Preservative.—(1) *Iodometric Method.**—Mix 10 cc. of the aldehyde solution (diluted if necessary to a strength not exceeding 3% of formaldehyde) with 25 cc. of tenth-normal iodine solution, and add drop by drop a solution of sodium hydroxide, till the color of the liquid becomes clear yellow. The solution is set aside for at least ten minutes, after which hydrochloric acid is added to set free the uncombined iodine, and the latter is titrated back with tenth-normal thiosulphate. Two atoms of iodine are equivalent to one molecule of formaldehyde, in accordance with the following reactions:

$$6NaOH + 6I = NaIO_3 + 5NaI + 3H_2O.$$
$$3CH_2O + NaIO_3 = 3CH_2O_2 + NaI.$$
$$5NaI + NaIO_3 + 6HCl = 6NaCl + I_6 + 3H_2O.$$

(2) *Method of Blank and Finkenbeiner.*†—Three grams of the solution are weighed into a tall Erlenmeyer flask, to which is then added from 25 to 30 cc. of twice-normal sodium hydroxide. 50 cc. of pure 2.5 to 3 per cent hydrogen peroxide solution are next gradually run in during a space of from three to ten minutes, through a funnel placed in the neck of the flask to prevent spurting, and the solution is allowed to stand for two or three minutes, after which the funnel is washed with water.

Finally the unused sodium hydroxide is titrated with twice-normal sulphuric acid, using litmus as an indicator. The less formaldehyde in the sample, the longer the mixture should stand after addition of the hydrogen peroxide, to complete the reaction. When less than 30% is present, it should stand at least ten minutes.

Ascertain the percentage of formaldehyde, by multiplying by 2 the number of cubic centimeters of soda solution used, when 3 grams of the sample are taken.

(3) *Ammonia Method.*‡—Weigh 10 grams of the formaldehyde solution into a flask, and treat with an excess of ammonia. Cork the flask and shake frequently during several days. The formaldehyde is by this process converted into hexamethylamine.

Transfer the solution to a tared platinum dish, and evaporate nearly

* Zeits. anal. Chem., 1897, 36, pp. 18–24; abs. Analyst, 22, p. 221.

† Ber., 31 (17), 2979.

‡ Conn. Exp. Sta., Annual Report, 1899, p. 143.

TIFICATION OF COLORS ON THE FIBER—*Continued.*

Character of Dyestuff.		Acid or Basic.	Hydrochloric Acid.		Sulphuric Acid.	
Dry.	Solution.		Fiber.	Solution.	Fiber.	Solution.
en metallic powder	Violet	Basic	Brown	Amber	Brown	Amber
ck powder	Violet	.....	Black	Colorless	Black	Blue
nze powder	Violet	.....	Violet	Blue	.....	Dark blue
.....	Indigo	.....	No change	.....	.....	Blue
eep-blue powder	Blue	.....	No action	.....	No action	
.....	.....	.....	Decolorized	.....	Decolorized	
nze powder	Blue	.....	Decolorized	Blue-green	Green	Green
ay-brown powder	Green	.....	Yellow	Red	Brown	Brown
en crystals	Bluish green	Basic	Orange	Orange	Bleached	Orange
ark-green powder	Green	.....	Yellow	Yellow	Olive	Drab

	Sodium Hydroxide.		Ammonia.		Spot with Nitric Acid.	Stannous Chloride.	Alcohol.
	Fiber.	Solution.	Fiber.	Solution.			
VIOLET-BLUE AND GREEN COLORS.							
Methyl violet	Decolorized	Colorless	Lilac	.....	.....	Green	Violet
Azo blue	Red	Pink	Dark violet	Red	.....	Decolorized	No action
Indulines soluble	.....	Violet decolorized by zinc dust	.....	AsNaOH	Dark green	Violet solution	Blue-violet
Fast blue R.							
Indigo	No change	.....	No change	.....	Yellow	Paler, solution yellow	Blue
Prussian blue *	Brown	.....	No action	.....	Green	No action	No action
Ultramarine blue †	No action	.....	No action	.....	.....	Decolorized	No action
Methylene blue ‡	Violet	.....	No action	.....	Green	Decolorized	Greenish blue
Resorcin green *	Darker	Greenish	No action	.....	Brown	Paler	No action
Malachite green	Decolorized	.....	Decolorized	.....	.....	Decolorized	Green
New Victoria green.							
New green.							
Naphthol green	Brighter	Olive	No action	.....	Orange	Decolorized	Pale blue

* Ash contains Fe. † Blue ash. ‡ $CaOCl_2$—decolorized.

ABBREVIATIONS OF FIRMS.

Ber. —Berlin Aniline Co.
D.H.—Durand, Huguenin & Co. Basle.
C. —L. Cassella & Co., Frankfort.
M. —Meister, Lucius & Brunig, Höchst.
By. —The Bayer Co., Elberfeld.
B.S.S.—Brooke, Simpson & Spiller, London.
P. —A. Poirier et G. Dalsace, Paris.
O. —K. Oehler, Offenbach.
H. —Read, Holliday & Sons, Huddersfield.
Sch. —The Schoellkopf Aniline and Chemical Co., Buffalo.

whole arrangement being such that a continuous current of methyl alcohol vapor bubbles through the liquid in the smaller flask, the heat being so regulated that from 15 to 25 cc. of methyl alcohol remains in the 150-cc. flask, while about 100 cc. of distillate passes into the receiving-flask in half an hour. Continue the distillation till all the acid has passed over, which is usually accomplished by distilling 100 cc. By a gentle aspiration upon the receiving-flask, loss by leaking may be avoided.

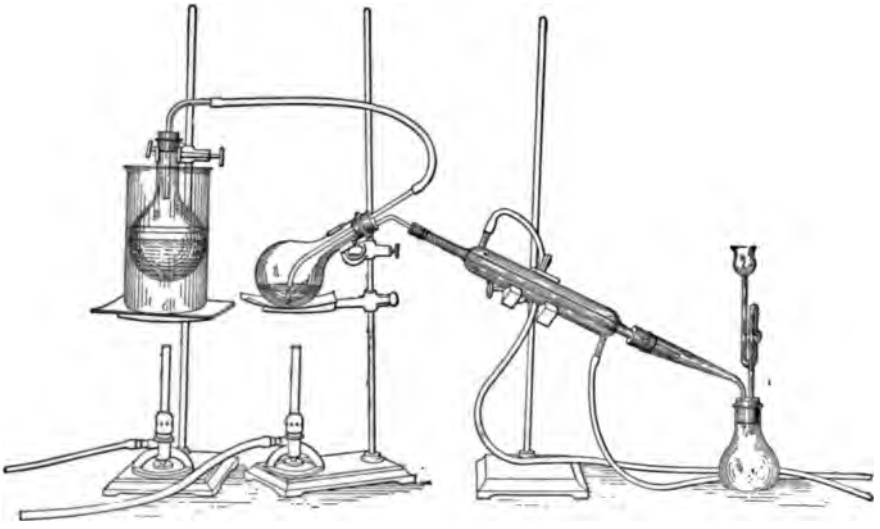


FIG. 117.—Apparatus for Determining Boric Acid According to Gladding.

Prepare a mixture of 40 cc. of glycerin and 100 cc. of water, and carefully neutralize, using phenolphthalein as an indicator. Add this mixture to the distillate, and titrate the whole with tenth-normal sodium hydroxide.

(1) *The Turmeric-paper Test.*—The most delicate test for boric acid, free or combined, is made by the aid of turmeric-paper, prepared by soaking a smooth, thin grade of filter-paper in an alcoholic tincture of powdered turmeric. The paper is afterwards dried and cut into strips, which are kept for convenience in a wide-mouthed bottle in a dark place.

Slightly acidulate the ash of the sample to be tested with a few drops of dilute hydrochloric acid, avoiding an excess of acid. Then dissolve the ash in a few drops of water and thoroughly saturate a strip of the turmeric-paper in the solution. On drying the paper, if boric acid either free or combined be present, a cherry-red coloration will be imparted to the paper, the depth of color depending on the amount present. As a confirmatory test, apply a drop of dilute alkali to the reddened paper, and a dark-olive color will be due to boric acid, sharply to be distinguished from the deep-red color produced when an alkaline solution is applied to ordinary turmeric-paper. The turmeric-paper reaction is delicate to 1 part in 8,000.

(2) *Tincture of Turmeric Test.*—To the solution to be tested, slightly acidified with hydrochloric acid, add an equal volume of saturated tincture of turmeric in an evaporating-dish, and heat for a minute or two. A red color, light or dark, depending on the amount of the preservative, is produced if boric acid be present, changed to an olive color by the addition of dilute alkali, after cooling.

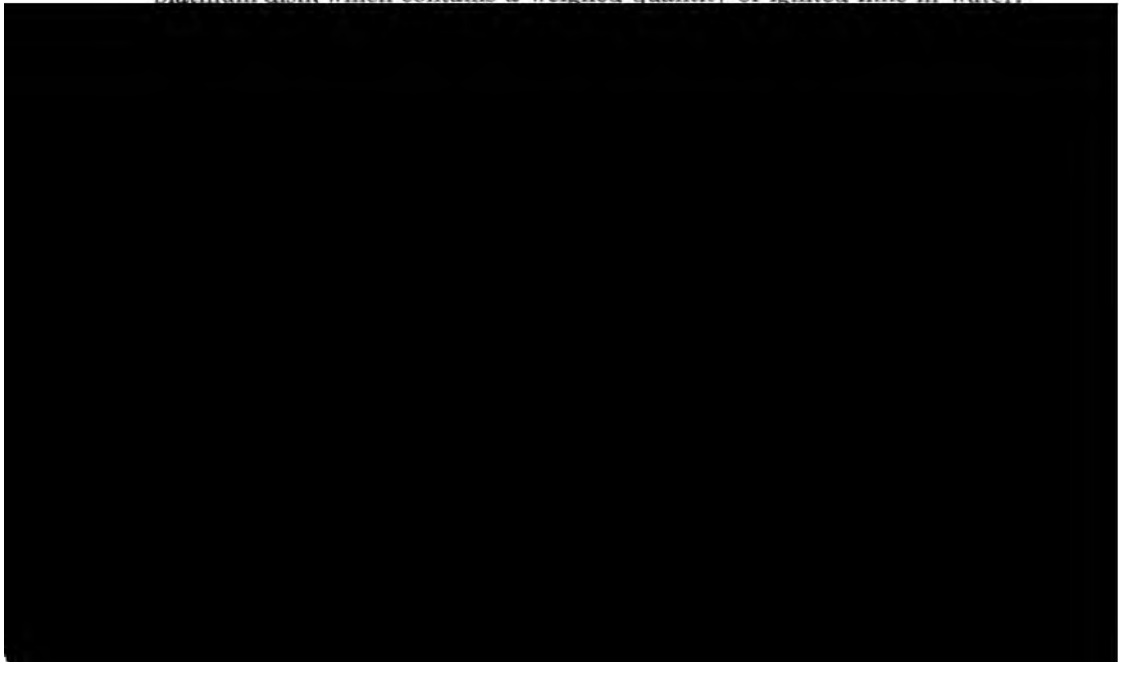
(3) *The Flame Test.*—A few cubic centimeters of alcohol are added to the dish containing the slightly acidulated ash of the sample to be tested, or to the acidulated dried residue from the evaporation of the aqueous solution of the suspected preservative, and after mixing by the aid of a stirring-rod, the alcohol is ignited. In the presence of any considerable portion of free or combined boric acid, a greenish tinge will be observed in the flame of the burning alcohol, especially at the first flash, due to the boric ether formed. This test is by no means as delicate at the paper test.

Determination of Boric Acid in Foods.—(1) *Thompson's Method.**—Add 1 or 2 grams of sodium hydroxide to 100 grams of the sample, and evaporate to dryness in a platinum dish. Char the residue thoroughly, and boil with 20 cc. of water, adding hydrochloric acid drop by drop till all but the carbon is dissolved. In burning, avoid too high a heat, simply charring sufficiently to insure a clear solution with water. Transfer by washing to a 100-cc. graduated flask, taking care that the volume does not exceed 50 or 60 cc. Add half a gram of dry calcium chloride, then a few drops

* Analyst, 18, p. 184.

of phenolphthalein solution, and next a 10% solution of sodium hydroxide, till a permanent pink color persists. Finally add 25 cc. of lime-water. By this means all phosphoric acid is precipitated in the form of calcium phosphate. Make up to the 100-cc. mark with water, shake, and pour upon a dry filter. To 50 cc. of the filtrate add sufficient normal sulphuric acid to remove the pink color. Then add a few drops of methyl orange, and continue the addition of sulphuric acid till the yellow is just turned to pink. Tenth-normal sodium hydroxide is then added * till the liquid takes on a faint yellow, excess of alkali being avoided. The salts of the acids present at this time are all neutral to phenolphthalein except boric acid and carbon dioxide. Boil the solution to expel the carbon dioxide, cool, add a little more phenolphthalein, and a quantity of glycerin equal in volume to the solution. Finally titrate with tenth-normal sodium hydroxide to a permanent pink color. Each cubic centimeter of tenth-normal sodium hydroxide equals 0.0062 gram crystallized boric acid, H_3BO_3, or 0.0035 gram boric anhydride, B_2O_3, or 0.00955 gram crystallized borax, $Na_2B_4O_7,10H_2O$.

(2) *Gooch's Method.*—Mix 400 to 500 grams of the substance with 10 grams of calcium hydrate, evaporate to dryness over a water-bath in a platinum dish, and burn cautiously to an ash. Dissolve the residue in cold nitric acid, and add an excess of silver nitrate to precipitate the chlorine. Filter, make up to 500 cc. with water, shake, and measure out 25 cc. into a 200-cc. flask fitted with a stopper provided with an outlet-tube, and with a separatory funnel forming virtually a thistle-tube, capable of being closed with a glass stop-cock. Through the outlet-tube connect the flask with a Liebig condenser provided with an adapter which can dip below the liquid in the receiver. As a receiver, use a 150-cc. tared platinum dish, which contains a weighed quantity of ignited lime in water.



SALICYLIC ACID.

Salicylic acid ($HC_7H_5O_3$) is a white, crystalline, strongly acid powder, made synthetically by treatment of carbolic acid with sodium hydroxide and carbon dioxide, or naturally from methyl salicylate (which occurs in oil of wintergreen to the extent of about 90%), by treatment of the wintergreen oil with strong potash lye. Most of the commercial salicylic acid is of the synthetic variety. Pure salicylic acid crystallizes from alcoholic solutions in 4-sided prisms, and from aqueous solution in long, slender needles. It melts at 155° to 156° C. It is slightly soluble in cold water (1 part in 450), and much more so in hot water. It is readily soluble in ether, alcohol, and chloroform.

It is frequently found on the market as a food preservative in the form of the much more soluble sodium salt, sodium salicylate, ($NaC_7H_5O_3$), which is, however, converted into salicylic acid when added to acid-fruit preparations, condiments, and liquors.

Sodium salicylate is a white, amorphous powder, soluble in 0.9 parts water and in 6 parts alcohol. It is prepared by treating salicylic acid with a strong, aqueous solution of sodium carbonate, and afterwards purifying. If a known weight of the powdered preservative be ignited, and a solution of the ash titrated with tenth-normal sulphuric acid, using cochineal as an indicator, each cubic centimeter of the acid is equivalent to 0.0160 gram of sodium salicylate.

Salicylic acid is largely used as a preservative of jellies, jams, and fruit preparations, canned vegetables, ketchups, table sauces, wines, beer, and cider. It is rarely used in milk and milk products, or in meats.

Bucholz has shown that 0.15% of salicylic acid is sufficient to prevent bacteria from developing in ordinary organic substances, while as small a quantity as 0.04% produces a marked restraining influence.

Detection of Salicylic Acid.—If the sample to be tested is of a similar nature to jelly, jam, ketchup, cider, etc., or capable of getting into aqueous solution, slightly acidify the liquid or pasty material, diluted, if necessary, with weak sulphuric (if not already acid), and shake directly with an equal bulk of ether, petroleum ether, or chloroform, in a corked flask, or in a separatory funnel. If the sample be too thick in consistency to shake directly, macerate in a mortar with alkaline water, and strain through cloth. Acidify the filtrate with dilute sulphuric acid, and then proceed to shake with the immiscible solvent as above. Separate by decantation or otherwise the immiscible solvent containing the preservative, if present, and

allow it to evaporate in an open shallow dish, either at room temperature or at a low heat. In case an emulsion forms on shaking, which is quite apt to happen, especially with ether for a solvent, divide the whole mixture between two tubes of a centrifuge of the form shown in Fig. 11, and whirl for three minutes at a high rate of speed. This usually serves to break up the most obstinate emulsion, so that it is easy to separate by decantation. If a considerable amount of salicylic acid be present, it will sometimes appear in the residue in the form of fibrous crystals.*

(1) To a portion of the dry residue add a drop of ferric chloride solution. A deep purple or violet color indicates salicylic acid.† If doubt exists as to the color, dilute with water, which often serves to bring out a distinctive purple coloration otherwise unobservable.

(2) Another portion of the residue may be heated with methyl alcohol and sulphuric acid. If salicylic acid be present, the well-known odor of methyl salicylate will be produced.

(3) A portion of the dry ether extract is warmed gently with a drop of concentrated nitric acid, and two or three drops of ammonia are added. Yellow ammonium picrate will be formed if a considerable quantity of salicylic acid be present, and a thread of wool free from fat may be dyed by soaking therein. This test is by no means as delicate as the ferric chloride color test.

Determination of Salicylic Acid.—A rough quantitative estimation of salicylic acid may be made colorimetrically, by carrying out more carefully the method already given for its detection with ferric chloride.

Make several extractions of a weighed or measured portion of the sample to be tested with ether or chloroform. Evaporate the combined extracts to dryness, and take up this residue with several washings of strong alcohol, which are filtered into a narrow graduated cylinder or tube. Treat with

BENZOIC ACID.

Benzoic Acid ($HC_7H_5O_2$) is produced by the oxidation of a large number of organic substances, particularly toluene. It is also extracted by sublimation from gum benzoin, which exudes from the bark of the *Styrax benzoin*, a tree growing in Java, Sumatra, Borneo, and Siam. Most of the commercial benzoic acid is made from toluene by treatment with chlorine and subsequent oxidation.

Benzoic acid crystallizes in leaflets, having a silky luster. It is odorless when cold, is soluble in 200 parts of cold, and 25 parts of boiling water, and readily dissolves in alcohol, ether, and chloroform. Its melting-point is 120°, and it sublimes at a slightly higher temperature.

Sodium Benzoate ($NaC_7H_5O_2$) is the salt most largely used in commercial preservatives, being much more soluble than the acid itself, into which, however, it is converted when put into acid fruit preparations. Sodium benzoate is prepared by adding benzoic acid to a concentrated hot solution of sodium carbonate till there is no longer effervescence, and then cooling, and allowing the sodium benzoate to crystallize out.

In titrating solutions of ignited sodium benzoate with tenth-normal sulphuric acid, each cubic centimeter of the standard acid is equivalent to 0.0144 gram of the benzoate.

Sodium benzoate is a white, amorphous powder, having a sweetish, astringent taste. It is soluble in 1.8 parts of cold water, and in 45 parts of alcohol. Benzoic acid is commonly found as a preservative of ketchups, jellies, jams, and canned goods, and less often in wines and liquors.

Detection of Benzoic Acid.—The sample is extracted with ether or chloroform in precisely the same manner as directed for salicylic acid. In fact, it is nearly always desirable to test the same sample for both these preservatives, since either and sometimes both are apt to be found in the same class of food products. For this purpose, the ether or chloroform extract is conveniently divided and evaporated to dryness in separate dishes, one of the residues to be tested for salicylic, and the other for benzoic acid.* A considerable amount of benzoic acid is apparent in the residue as shining crystalline scales or needles.

(1) *Ferric Chloride Test.*—A portion of the residue from the ether extract is dissolved in ammonia, and evaporated to dryness over the water-bath. The residue is stirred in a few drops of warm water, and the latter

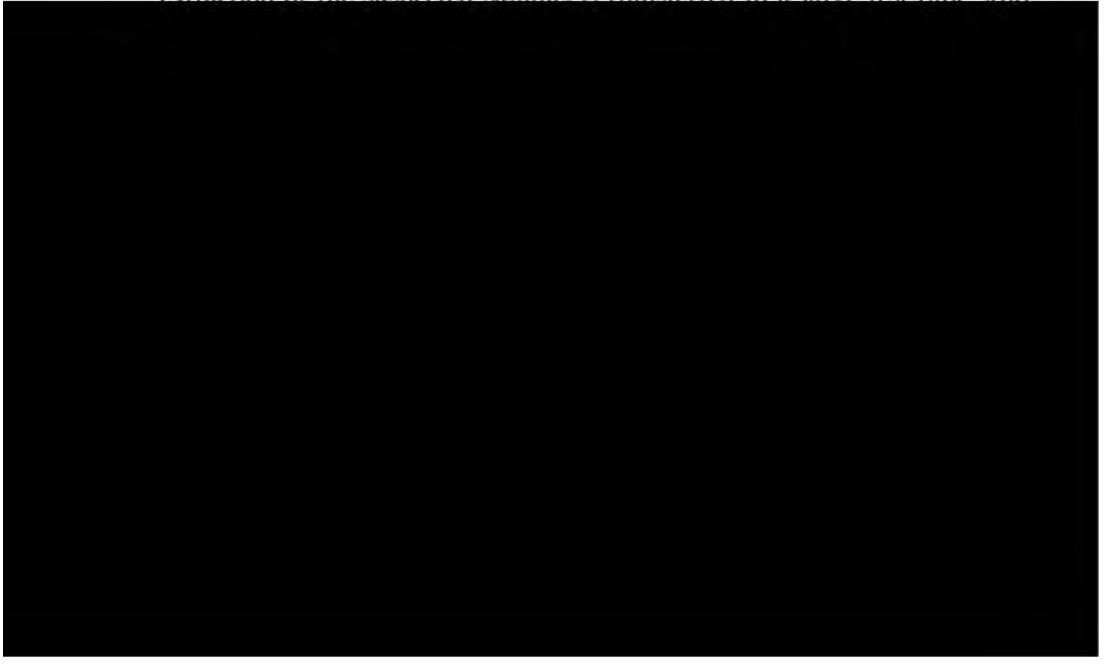
* See also method on p. 142.

solution is poured through a filter, and collected in a narrow test-tube. A drop of ferric chloride is added, and in the presence of benzoic acid a flesh-colored precipitate of ferric benzoate is produced, very characteristic and unmistakable, because of its peculiar color, when the solution in which the test is made is colorless. It occasionally happens, however, in the case of jellies, jams, and ketchups, that these preparations are artificially colored with a dyestuff that persists by its depth of color in obscuring that of the ferric benzoate, especially when only a small amount of benzoic acid is present. Again, in such products as sweet pickles, a precipitate of basic ferric acetate might also come down with the ferric benzoate, and thus confuse. In such cases one of the following methods should be carried out.

(2) *Sublimation Method.**—Evaporate an ammoniacal solution of the ether extract to dryness in a large watch-glass, by the aid of a gentle heat. Fasten with clips or otherwise a second watch-glass, to the first, edge to edge, so as to form a double convex chamber, with a cut filter-paper between. Place upon a small sand-bath and heat. Benzoic acid, if present, will sublime upon the surface of the upper glass in minute needles, recognizable under the microscope. It may further be tested by determining the melting-point of the crystals, or by treating the residue with ammonia, and after evaporation and solution of the residue, applying the ferric chloride test as above.

(3) *Peter's Oxidation Method.*†—This method depends on the oxidation of benzoic to salicylic acid by the action of sulphuric acid and barium peroxide, and should, of course, be applied only when salicylic acid has been first proved absent.

A portion of the residue, say 0.1 gram, from the ether or chloroform extraction of the suspected sample is transferred to a large test-tube and



cooled, and filtered. The filtrate is then extracted with ether or chloroform, and the extract tested in the regular manner for salicylic acid.

Mohler's Test for Benzoic Acid.*—The ether extract or the distillate from the suspected sample is evaporated to dryness, and the residue treated with 2 or 3 cc. of strong sulphuric acid. Heat till white fumes appear; organic matter is charred and benzoic acid is converted into sulpho-benzoic acid. A few crystals of potassium nitrate are then added. This causes the formation of metadinitrobenzoic acid. When cool, the acid is diluted with water, and ammonia added in excess, followed by a drop or two of ammonium sulphide. The nitrocompound becomes converted into ammonium metadiamidobenzoic acid, which possesses a red color. This reaction takes place immediately, and is seen at the surface of the liquid without stirring.

SULPHUROUS ACID AND THE SULPHITES.

Free sulphurous acid is not often, if ever, applied as such to foods, but from the "sulphuring" process, to which wine casks are often subjected, the free acid is sometimes found in the wine. The sulphurous acid salts most commonly employed as food preservatives are the bisulphites of sodium and calcium, $NaHSO_3$ and $Ca(HSO_3)_2$. Others used to some extent are the normal sodium sulphite, and also potassium and ammonium sulphite. The sulphites are usually commercially prepared by passing sulphurous acid gas through strong solutions of the carbonates. Acid sulphites are formed by an excess of the sulphurous acid in the solution of the sulphite. The acid sulphites are distinguishable from the sulphites by their reaction with litmus paper, the former being acid, while the latter are neutral or feebly alkaline. All of these salts have a bitter, salty, and highly sulphurous taste, and possess a very pungent, irritating odor. With the exception of normal calcium sulphite, all of the above are readily soluble in water.

The sulphites are most commonly used as preservatives of fruit juices, ketchups, fruit and vegetable pulps, and meat products. They are frequently mixed with other antiseptics, as with the salts of salicylic and benzoic acids.

Detection of Sulphurous Acid.—Mix 100 or 200 grams of the sample (finely macerated in a mortar if necessary) with enough water to form a thin solution or paste. Acidify with phosphoric acid, transfer to a flask, and subject to distillation. The delivery-tube from the condenser should

* Bul. Soc. Chim., 1890. 3, 414; U. S. Dept. of Agric., Bur. of Chem., Bul. 65, p. 109.

preferably dip below the surface of a few cubic centimeters of water in the receiver. Distill off 20 or 30 cc., treat the distillate with a few drops of bromine water, and boil. Then add a little barium chloride reagent. A precipitate indicates sulphurous acid.

Determination of Sulphurous Acid.*—To 25 grams of the sample, finely divided in water, if solid or semi-solid, add 25 cc. of a normal solution of potassium hydroxide in a 200-cc. flask. Shake thoroughly, and set aside for at least fifteen minutes with occasional shaking. 10 cc. of sulphuric acid (1:3) are then added with a little starch solution, and the mixture is titrated with N/50 iodine solution, introducing the iodine solution quite rapidly, and adding it till a distinct fixed blue color is produced. 1 cc. of the iodine solution is the equivalent of 0.00064 gram SO_2.

FLUORIDES, FLUOSILICATES, AND FLUOBORATES.

These substances all possess strong antiseptic qualities, and while no instances are recorded of the use of the last two classes of compounds in this country, the use of fluorides as a preservative of beer is practiced to some extent. The salt most commonly used is ammonium fluoride (NH_4F), preparations of this salt being sold commercially under various trade names as beer preservatives. Ammonium fluoride exists as small, deliquescent, hexagonal, flat crystals. Its taste is strongly saline. It is soluble in water, and slightly soluble in alcohol. Sodium fluoride (NaF) occurs as clear, lustrous crystals, soluble in water.

Detection of Fluorides.†—One hundred grams or so of the beer or other solution to be tested are made alkaline with ammonium carbonate and heated to boiling. The fluorine is then precipitated by the addi-



be found etched upon the glass. Small amounts of silica impair the delicacy of the test.

Detection of Fluoborates and Fluosilicates.*—Two hundred cc. of the wine or other sample are made alkaline with lime water, evaporated to dryness, and ignited. The crude ash is first extracted with water acidified with acetic acid, and the solution filtered. The insoluble residue is again ignited and extracted with dilute acetic acid, which is filtered off and added to the first extract. The filtrate contains the boric acid, if present, and this is tested for as directed on page 669. Calcium silicate or fluoride, if present, is in the insoluble portion.

Incinerate the filter with the insoluble portion, transfer the ash to a test-tube, mix with some silica, and add a little concentrated sulphuric acid. A small U-tube should be attached to the test-tube, containing a very little water. The test-tube is immersed for half an hour in a beaker of water kept hot on a steam-bath. In the presence of fluoride, silicon fluoride will be generated, and will be decomposed by the water, forming a gelatinous deposit on the walls of the tube.

If both boric and hydrofluoric acids are found, the compound present is undoubtedly a borofluoride. If no boric acid is found, but silicon fluoride is detected, repeat the operation, but without the added silica. If the silicon skeleton is then formed, fluosilicate is probably present.

BETA-NAPHTHOL.

Beta-naphthol ($C_{10}H_7OH$) is a phenol, occurring naturally in coal-tar, but the commercial product is more commonly prepared artificially from naphthalene by digesting the latter with sulphuric acid, and fusing the product with alkali. It is a colorless, or pale buff-colored powder, with a faint phenolic odor and a sharp taste. It is slightly soluble in water, and readily soluble in alcohol, ether, and chloroform. Its melting-point is 122° C. In alcoholic solution it is neutral to litmus.

It is used to some extent in alcoholic solution as a preservative of cider.

Detection of Beta-Naphthol.—*Bube* † states that if an ethereal extract of beta-naphthol is evaporated to dryness, and the residue dissolved in hot water made first faintly alkaline with ammonia, and then faintly acid with very dilute nitric acid, a beautiful rose color will be developed on the addition of a drop of fuming nitric acid or of a nitrite. He declares

* U. S. Dept. of Agric., Bur. of Chem., Bul. 59, p. 63.

† Analyst, 13 (1888), p. 52.

the test to be a delicate one, but it is apparently sometimes obscured by interfering substances, which the ether may dissolve. It should also be carried out in a faint light, as strong sunlight affects the color.

Ferric chloride, when applied to an aqueous solution of beta-naphthol, produces a greenish coloration.

Shake about 50 grams of the sample to be tested with chloroform in a separatory funnel, evaporate the chloroform extract to a small volume (say 1 or 2 cc.), transfer to a test-tube, add 5 cc. of an aqueous solution of potassium hydroxide (1:4), and warm gently. If beta-naphthol is present, a deep-blue color will appear in the aqueous layer, turning through green to light brown.

ASAPROL, OR ABRASTOL.

These are trade names for calcium α-mono-sulphonate of beta-naphthol, $Ca(C_{10}H_6SO_3OH)_2$, a white, odorless, scaly powder, sometimes slightly reddish, obtained by the action of heated sulphuric acid on beta-naphthol, the resulting compound being afterwards treated with a calcium salt. It is readily soluble in water and alcohol, and is neutral in reaction. Its taste is at first slightly bitter, but rapidly changes to sweet. It decomposes at about 50° C.

The writer is unaware of any instance of the presence of this substance in foods, but its character is such as to adapt it for use as a preservative of wines and possibly other food products. It has long been regarded as a possible preservative, and the analyst should be prepared to encounter it at any time.

Detection of Asaprol.—*Sinabaldi's Method.**—The portion of the



green precipitate appears, add 5 cc. of alcohol, dissolve in sulphuric acid, shake, and filter. If abrastrol be present to the extent of 0.01 gram or more, a red coloration is observed, while in its absence, the filtrate is colorless or faintly yellow.

If the solution to be tested is a fat, it should be melted and extracted with hot 20% alcohol, which is evaporated to dryness, and the above test carried out on the dry residue.

REFERENCES ON PRESERVATIVES AND THEIR USE IN FOOD.

ABEL, R. Zum Kampfe gegen die Konservierung von Nahrungsmitteln durch Antiseptica. Hyg. Runds., 1901, 265-281.

ANNETT, H. E. Boric Acid and Formaline as Milk Preservatives. Thompson Yates Lab. Reports, Liverpool, Vol. II, 1900, pp. 57 67.

BALDWIN, H. B. Toxic Action of Sodium Fluoride. Jour. Am. Chem. Soc., 21, 1899, p. 517.

BENEDICENTI. Action of Formaldehyde on Various Proteid Substances. Archw. f. Anat. u. Physiolog., 1897, p. 219.

BISCHOFF, H., and WINTGEN, U. Beiträge zur Konservenfabrikation. Ztsch. für Hyg., Bd. 34, 1900, Heft 3, 496–513.

BLISS and NOVY. Action of Formaldehyde on Enzymes. Jour. Exp. Med., 4, 47.

CHITTENDEN, R. H. Influence of Borax and Boracic Acid on Digestion. Diet. and Hyg. Gazette, 9, 1893, 25.

CHITTENDEN and GIES. Effects of Borax and Boric Acid on Nutrition. New York Med. Jour., Feb., 1898.

—— Experiments with Borax and Boric Acid on the Lower Animals. Am. Jour. of Phys., Vol. I, No. 1, 1898.

DIGHT, C. F. Effect of Boric Acid and Borax on the Human Body, and with Particular Reference to their Use as Food Preservatives. Minneapolis, 1902.

GOUIN, R. Le Beurre et l'Acide Borique. Jour. d'Agricult prat., 1900, p. 14-16.

GRUBER. Ueber die Zulässigkeit der Verwendung der Fluoride zur Konservierung von Lebensmittel. Das Oesterr. Sanitätsw., 1900, 4.

—— Ueber die Zulässigkeit der Verwendung von Chemikalien zur Konservierung von Lebensmittel. Das Oesterr. Sanitätsw., 1900.

GRÜNBAUM, A. S. Note on the Value of Experiments in the Question of Food Preservatives. Brit. Med. Jour., 1901, p. 1337.

HALLIBURTON, W. D. Remarks on the Use of Borax and Formaldehyde as Preservatives of Food. Brit. Med. Jour., 1900, pp. 1–2.

HEFFTER, A. Ueber den Einfluss der Borsäure auf die Ausnutzung der Nahrung. Arbeiten aus dem kaiserlichen Gesundheitsamte, Bd. 90, Part 1, 1902, p. 97.

HILL, A. Antiseptics in Food. Pub. Health Jour., London, 11 (1901), 527.

HOPE, E. W. Preservatives and Coloring Matters in Foods. Thompson Yates Lab. Reports, Vol. III (1900), pp. 75–78.

KISTER, J. Ueber Gesundheitschädlichkeit der Borsäuer als Konservierungsmittel für Nahrungsmittel. Zeit. f. Hygiene, Bd. 37, 1901, Heft 2, p. 225.

LAUGE, L. Beiträge zur Frage der Fleischkonservierungmittel. Borsäure, Borax und Schwefeligsauren Natronzusätzen. Mit einem Anhang. Milchkonservierung betr. Arch. f. Hygiene, Bd. 40, 1901, 2, pp. 143–186.

LEBBIN, G. Die Konservierung und Färbung von Fleischwaaren. Hyg. Rund., 11, No. 23.

LEBBIN u. KALLMANN. Ueber die Zulässigkeit Schwefeligsauer Salze in Nahrungsmitteln. Zeits. für öffentl. Chem., 7, 17, 324–334.

LEBBIN, G. Preservation and Coloring of Meat Produce. Translated from the German.

—— Should the Use of Boric Acid as a Food Preservative be Permitted? Translated from the German of Die medicinische Woche, Sept., 1901.

LEFFMANN, H. Food Preservatives. Penn. Board of Agric., An. Rep., 1897, 535.

—— Influence of Preservatives on Digestive Enzymes. Diet. and Hyg. Gazette, 14, 718.

—— Hygienic Relations of Boric Acid and Borax. Diet. and Hyg. Gazette, 14, 171.

—— Digestive Ferments and Preservatives. Jour. Frankl. Inst., 147 (1899), 97.

LEPIERE. Action of Formaldehyde on Proteids. Bul. Soc. Chem., 21 (1899), p. 729.

LIEBREICH, O. Effects of Borax and Boric Acid on the Human System. London, 1902.

—— The So-called Danger from the Use of Boric Acid in Preserved Foods. Lancet, 1900, p. 13–15.

LOEW. Action of Formaldehyde on Pepsin and Diastase. Jour. f. prakt. Chem., 1888, 37, p. 101.

NEUMANN, R. O. Ueber den Einfluss des Borax auf dem Stoffwechsel des Menschen. Arbeiten aus dem kaiserlichen Gesundheitsamte, Bd. 90, Pt. 1, 1902, p. 89.

POLENSKE. Ueber den Borsäuregehalt von frischen und geräucherten Schweineschinken. Loc. cit., 167.

RIDEAL, S. Formalin as a Milk Preservative. Analyst, 20, 157.

—— Disinfection and the Preservation of Food. London and New York, 1903.

—— On the Use of Boric Acid and Formic Aldehyde as Milk Preservatives. Public Health Jour., London, 11, 1901, 554.

ROHARDT, W. Ueber Konservierung von frischem Fleisch und ueber Fleischkonserven von Hygienischen- und Sanitäts-polizeilichem Standpunkt aus. Vierteljahresschrift f. gerichtl. Med., 1901, Heft 2, p. 321.

ROST, E. Ueber die Wirkungen der Borsäure und des Borax auf den thierischen und

VAUGHAN, V. C., and VEENBOER, W. H. The Use of Boric Acid and Borax as Food Preservatives. Am. Medicine, March, 1902.

VAILLARD, L. Les Conserves alimentaires de Viande. Rev. d'Hyg., 1900, pp. 782–792.

WEITZEL, A. Ueber die Labgerinnung der Kuhmilch unter dem Einfluss von Borpräparaten und anderen chemischen Stoffen. Arbeiten aus dem kaiserlichen Gesundheitsamte, Bd. 90, Part 1, 1902, p. 126.

REPORT OF THE DEPARTMENTAL COMMITTEE appointed to Inquire into the Use of Preservatives and Coloring Matters in the Preserving and Coloring of Food. 497 pp. London.

CHAPTER XVIII.

ARTIFICIAL SWEETENERS.

UNDER this head are included the intensely sweet coal-tar derivatives, such as saccharin, dulcin, and glucin, that possess no food value whatever in themselves. From their high sweetening power, in some cases several hundred times that of cane sugar, they are capable, when used in minute quantity, of imparting an appropriate degree of sweetness to food products, which, on account of the use of inferior materials, or by reason of the presence of inert or less sweet adulterants, would otherwise be lacking in this property.

Such canned vegetables as sweet corn and peas are subject to treatment with saccharin, especially if by their age and condition before canning they are wanting in the sweet, succulent taste inherent in the fresh product.

The sweetening power of commercial glucose is considerably less than that of cane sugar, so that when large admixtures of the glucose are used in such products as jellies, jams, honey, molasses, maple syrup, etc., to the exclusion of cane sugar, the presence of the glucose might in some cases be suggested by the bland taste of the food, unless reinforced by one of the artificial sweeteners.



pure, is about 224° C. It is prepared from toluene, which by treatment with concentrated sulphuric acid is first converted into a mixture of ortho- and para-toluene sulphonic acids. These are further converted into corresponding chlorides, and from the orthochloride, by treatment with ammonia, the imide is formed. It is soluble in 230 parts of cold water, 30 parts of alcohol, and 3 parts of ether. It is sparingly soluble in chloroform, but readily soluble in dilute ammonia. It is from 300 to 500 times as sweet as cane sugar, and, unlike cane sugar, it is not, when pure, charred by the action of concentrated sulphuric acid even on heating. Its aqueous solution is distinctly acid in reaction. Pure saccharin, when heated under diminished pressure, can be sublimed without decomposition.

The addition of 1 part of saccharin to 1,000 parts of commercial glucose renders the latter as sweet as cane sugar.

A sodium salt of saccharin is found on the market, prepared by neutralizing a solution of saccharin with sodium hydroxide or carbonate. The sodium salt crystallizes in the form of rhombic plates, forming a white powder readily soluble in water, and possessing nearly the same sweetening power as saccharin. It is sometimes put up in the form of tablets for the use of diabetic patients as a substitute for sugar.

Saccharin, aside from its sweet taste possesses, according to Fahlberg and List,* antiseptic properties, and on this account it is sometimes used in beer and other liquors. Squibb states that saccharin has about the same power as boric acid as an antiferment

Detection of Saccharin in Foods.—If the sample to be tested is a solution or syrup, render it acid, if not already such, with phosphoric acid, and extract with ether. In case of canned vegetables and similar goods, finely divide the material by pulping or maceration in a mortar, dilute with water, and strain through muslin. Acidify the filtrate, and extract with ether.† If an emulsion forms, use a centrifugal machine (p. 23). Separate the extract, evaporate off the ether, and test the residue for saccharin as follows:

(1) Add to the residue, if it tastes sweet, a few cubic centimeters of hot water, or preferably a very dilute solution of sodium carbonate, in which saccharin is more soluble. An intensely sweet taste is indicative of its presence. This test, if applied directly, will sometimes fail, especially in the case of beer, by reason of the extraction by the ether of various

* Jour. Soc. Chem. Ind., IV, p. 608.

† Allen states that a purer residue is obtained if the sample of beer be treated with lead acetate, and filtered before extraction with ether.

bitter principles, such as hop resins, which by their strong, bitter taste mask the sweet taste of saccharin in the residue. Spaeth * recommends that such bitter substances be removed before extraction, which is done by treatment of 500 cc. of the beer with a few crystals of copper nitrate, or with a solution of copper sulphate. The flocculent precipitate formed need not be filtered off, but the liquid is preferably concentrated by evaporation to syrupy consistency, acidified with phosphoric acid, and extracted with three successive portions of a mixture of ether and petroleum ether. After extraction, separation, and evaporation of the solvent, dissolve the residue in weak sodium carbonate. As small a quantity as 0.001% of saccharin can be detected in the final alkaline solution by its sweet taste.

(2) *Bornstein's Test.*†—Heat the residue from the ether extraction of the acidified sample with resorcin and a few drops of sulphuric acid in a test-tube till it begins to swell up. Remove from the flame, and, after cooling till the action quiets down, again heat, repeating the heating and cooling several times. Finally cool, dilute with water, and neutralize with sodium hydroxide. A red-green fluorescence indicates saccharin. Gantter ‡ states that it is useless to apply this test to beer, in view of the fact that ordinary hop resin gives the same fluorescence.

(3) *Schmidt's Test.*§—The residue is heated in a porcelain dish with about a gram of sodium hydroxide ‖ for half an hour at a temperature of 250° C., either in an air-oven or in a linseed oil bath. This converts the saccharin if present into sodium salicylate. Dissolve the fused mass in water, acidify, and extract the solution with ether. Test the ether residue in the regular manner for salicylic acid with ferric chloride (p. 672). This test can obviously be applied only in the absence of



this principle depends the following method of Reischauer:* A known quantity of the beer or other liquid to be tested is concentrated by evaporation to about one-third its original volume, acidified with phosphoric acid, and extracted by repeated portions of ether. The combined ether extract is evaporated to small volume, and transferred to a platinum crucible, in which it is further brought to dryness. It is then cautiously ignited with a mixture of about 6 parts sodium carbonate and 1 part potassium nitrate. Dissolve the fusion in water, acidulate with hydrochloric acid, and determine the sulphuric acid in the usual manner with barium chloride. The weight of the precipitated barium sulphate, multiplied by 0.785, gives the weight of saccharin. In view of the fact that only small quantities of saccharin are used in beer and other foods, it is best to employ a large portion of the sample for analysis.

DULCIN.

Dulcin or sucrol, para-phenetol carbamide ($C_2H_5O.C_6H_4.NH.CO.NH_2$) is a white powder, composed of needle-like crystals, sparingly soluble in cold water, ether, petroleum ether, and chloroform. It dissolves in 800 parts of cold water, 50 parts of boiling water, and 25 parts of 95% alcohol. It is readily soluble in acetic ether. Its melting-point is about 173° C. It is not readily sublimed without decomposition. Dulcin is about four hundred times sweeter than cane sugar.

When a mixture of dulcin and dilute sodium hydroxide is subjected to distillation, phenetidin goes over with the steam into the distillate. When this is heated with glacial acetic acid, phenacetin is formed, which may be tested for as follows: Boil with hydrochloric acid, dilute with water, cool, filter if turbid, and add a few drops of a solution of chromic acid. A deep-red color indicates phenacetin.

Detection of Dulcin in Foods.—In view of the comparatively slight solubility of dulcin in ether and chloroform, acetic ether is the best solvent for purposes of removing it from foods, first making it alkaline.

(1) *Bellier's Method.*† —A portion of the sample to be tested is made alkaline and extracted with acetic ether. In the case of certain products it is best to subject them to varied preliminary treatment, depending on the case in hand. With such products as thin fruit syrups, simply make alkaline and shake out with acetic ether. In the case of thick fruit syrups, confectionery, and preserves, dilute with water, add an excess of basic

* Abst. Analyst, 11, p. 234.

† Ann. de Chim. Anal., 1900, V, pp. 333–337; Abs. Analyst, 26, p. 43.

lead acetate, remove the lead by precipitation with sodium sulphate, filter, and make the filtrate alkaline.

With wines, add 2 grams of mercuric acetate and a slight excess of ammonia, shake, and filter.

With beer, add to 200 cc. 2 or 3 grams of powdered sodium phosphotungstate, and a few drops of sulphuric acid, shake, allow to stand for a few minutes, and filter. Make the filtrate alkaline with ammonia.

Having thus obtained a clarified solution, use from 50 to 200 cc. of neutral acetic ether to say 500 cc. of the alkaline solution, and shake in a separatory funnel. Separate the extract, filter, and evaporate to dryness. If the dulcin exceeds 0.04 gram per liter, crystals will be apparent in the residue. If fats and resins are present in the residue, make repeated extractions with hot water, and evaporate to dryness. The purified residue is finally brought to dryness in a porcelain dish, and treated with 1 or 2 cc. of sulphuric acid and a few drops of a solution of formaldehyde. Let it stand for fifteen minutes, and afterwards dilute with 5 cc. of water. A turbidity or precipitate indicates dulcin.

(2) *Jorissen's Test.**—The residue from the acetic ether extract of an alkaline solution of the sample is treated with 2 or 3 cc. of boiling water in a test-tube, and a few drops of mercuric nitrate † are added. Heat the tube and its contents for five minutes in a boiling water-bath, withdraw, and disregarding any precipitate, add a small quantity of lead peroxide. On the subsidence of the precipitate, which quickly occurs, a fine violet color appears for a short time in the clear upper layer in presence of 0.001 gram of dulcin.

(3) *Morpurgo's Method.*‡—To the acetic ether residue, evaporated to dryness in a porcelain dish, add a few drops of phenol and concentrated sulphuric acid, and heat a few minutes on the water-bath. After cooling,



evaporation, and make up to the original volume with water. Treat the various food preparations with the appropriate clarifying reagents, as in Bellier's qualitative test (p. 685), and, after filtering and making alkaline, extract twice with 50 cc. each of acetic ether. The residue is purified if necessary by extraction with hot water as above described, and the final residue is dissolved in 1 to 5 cc. of concentrated sulphuric acid. A few drops of formaldehyde are added. The solution is allowed to stand for fifteen minutes, and then diluted to ten times its volume with distilled water. After twenty-four hours, collect the precipitate on a tared filter, wash with water, dry, and weigh.

GLUCIN.

This comparatively new sweetening agent is the sodium salt of a mixture of the mono- and di-sulphonic acids of a substance having the composition $C_{19}H_{16}N_4$. In the market it appears as a light-brown powder, readily soluble in water. It is insoluble in ether and chloroform. It decomposes without melting at about 250° C. It is three hundred times sweeter than cane sugar.

A color reaction with glucin is obtained by dissolving it in dilute hydrochloric acid, cooling by immersing the test-tube in water, and to the cold solution adding a little sodium nitrite solution. Finally, to the liquid is added a few drops of an alkaline solution of beta-naphthol, and a red coloration is produced. With resorcin or salicylic acid in alkaline solution, the color will be yellow.

REFERENCES ON ARTIFICIAL SWEETENERS.

ALLEN, A. H. The Detection of Saccharin in Beer. Analyst, 13 (1888), p. 105.

BELLIER, J. The Detection and Estimation of Dulcin in Beverages. Ann. de Chem. Anal., 1900, 5, 333; Abs. Analyst, 26, 1901, p. 43.

BERLIOZ, F. Influence of Saccharin upon Digestion. Chem. Zeit., 1900, p. 416; Abs. Analyst, 25, 1900, p. 233.

BUCHKA, K. V. Künstliche Süssstoffe. Vereinbarung. v. Nahr. u. Genuss. f. d. deuts. Reich., Heft II, p. 134. Berlin, 1899.

COHN, G. Ueber künstliche Süssstoffe. Apoth. Ztg., 1898, 13, pp. 796 and 804.

DEFOURNEL, H. Determination of Saccharin in Food Products. Abs. Analyst, 26, 1901, p. 268.

DENNHARDT. Versuche zum Nachweise des Dulcins. Ber. d. deutsch. pharm. Ges., 1898, 6, p. 287.

GANTTER, F. The Detection of Saccharin in Beer. Abs. Analyst, 18, 1893, p. 184.

GRAVILL, E. D. Notes on Saccharin. Pharm. Jour., 8, 1887 (3), pp. 18, 337.

HERZFELD, A., and WOLFF, F. Ueber die Bestimmung der künstlichen Süssstoffe in Nahrungsmitteln. Zeits. f. Unters. d. Nahr. u. Genussm., 1898, 1, p. 839.

JORISSEN, A. Neue Methode zum Nachweise von Dulcin in Getränken. Jour. de Pharm. de Liège; Chem. Centr., 1896, 1, p. 1084; Abs. Analyst, 21, 1896, p. 164.

LEYS, A. A New Test for Saccharin. Ann. de. Chim. anal., 1901, 6, p. 201; Abs. Analyst, 26, 1901, p. 321.

REID, E. E. Valuation of Saccharin. Am. Chem. Jour., 1899, 21, p. 461.

RUGER, C. Ueber das Fahlberg'sche Saccharin. Gesundheit, 1888, 13, p. 241.

SCHMITT, C. Ueber den Nachweis der o. Sulfamin-benzoesäuer, genannt "Fahlberg'sches Saccharin." Rep. für anal. Ch., 1887, 7, p. 437.

SPÄTH, E. Ueber den Nachweis des Saccharins im Bier. Zeits. f. angew. Chemie, 1893, p. 579.

SUTHERLAND, D. A. Saccharin. Jour. Soc. Chem. Ind., 6, 1887, p. 808.

TAYLOR, W. A. H. Commercial Saccharine. Pharm. Jour., 1887, 88 (3), 18, 377.

THOMS, H. Ueber Dulcin. Ber. d. deutsch. pharm. Gesellsch., 1893, 3, 133.

WANTERS, J. Nachweis des Saccharins im Bier. Moniteur scientifique, 4, 1896, 10, 146.

CHAPTER XIX.

CANNED AND BOTTLED VEGETABLES, RELISHES, AND FRUIT PRODUCTS.

CANNED VEGETABLES AND FRUITS.

STRICTLY speaking all varieties of canned foods found in the market, whether meats, fruits, or vegetables, in order to be entirely beyond criticism, should not differ from the corresponding freshly cooked varieties which they are intended to replace, excepting that they are free from bacteria. Such a degree of perfection is, however, difficult, even if possible, to attain, and nearly all commercial canned products, even if made from the best materials, are liable to contain either antiseptic substances or coloring-matter intentionally added by the manufacturer, or metallic impurities accidentally derived from the vessels in which they are prepared, or from the containers in which they are sealed. In spite of these objections, canned foods form a convenient, and in some cases indispensable means of furnishing both necessities and luxuries for the table. The canning of foods is especially useful for preserving them during long periods of time, for enabling certain fruits and vegetables to be enjoyed out of season, and for furnishing supplies in a convenient manner to inaccessible places where fresh foods are not readily obtainable, as in the case of armies in the field, of vessels at sea, of campers in the woods, etc. Canned goods in great variety are used in nearly every household.

When it is considered that in the United States alone something like one hundred million cans of corn are packed in a single year, about the same quantity of peas, and one hundred and fifty million cans of tomatoes, to say nothing of an ever-increasing variety of other foods, some idea may be gained of the enormous proportions to which the canning industry has grown. It is comforting to know that, in view of their wide-spread consumption, the greater portion of such foods found on the market are

comparatively harmless, as is evidenced by the fact that few cases of injury to health have been directly traceable to their use.

Method of Canning Food.—Various modifications as to details exist with different products and in different localities, but in general the principle of canning in tin is the same in all cases. The fresh product is cleaned carefully, and packed in cans with the requisite amount of water. The cans are then sealed, and subjected to the effect of steam or boiling water till the contents are thoroughly cooked. Each can is then tapped or punctured at one end to expel the air, and again heated, after which the hole is closed by a lump of solder, thus forming a vacuum in the can, which is afterwards heated for a sufficient time to destroy the bacteria, usually for several hours.

The above mode of procedure is the time-honored one, and is still in vogue in most localities, but a more modern method, much in use at present, consists in first cooking the food at a temperature of 82° to 88° C. before transferring to the cans, and afterwards subjecting the cans when sealed to a high heat of about 125° C. in dry air in so-called retorts, this heating or "processing," as it is termed, being carried on for a sufficient length of time to completely sterilize the contents of the can. Obviously a much shorter time is required for this than when the temperature of boiling water is employed, and the sterilization is much more effective.

Cooked vegetables and fruit products put up in glass jars or bottles are tightly sealed when hot, either with screw-caps or with some form of cover held by a clamp, or with metal or hard-rubber caps fitting over a flanged mouth. Frequently a soft-rubber ring is inserted between the cover and the mouth of the jar or bottle. The material of the cover is generally either glass, porcelain, or metal. Cork stoppers are, however, sometimes pressed into the mouths of the bottles and made extra tight



the ends of the can are usually concave. The highest aim of the canner should be to retain in his product as far as possible the appearance, palatability, and nutritive value of the freshly cooked food.

PROXIMATE COMPOSITION OF CANNED VEGETABLES AND FRUITS.*

	No. of Analyses.	Water.	Protein.	Fat.	Total Carbohydrates.	Crude Fiber.	Ash.	Fuel Value per Pound.
CANNED VEGETABLES.								
Artichokes	3	92.5	.8	.....	5.0	.6	1.7	110
Asparagus	14	94.4	1.5	.1	2.8	.5	1.2	85
Beans, baked	21	68.9	6.9	2.5	19.6	2.5	2.1	600
" string	29	93.7	1.1	.1	3.8	.5	1.3	95
" Lima	16	79.5	4.0	.3	14.6	1.2	1.6	360
Brussels sprouts	1	93.7	1.5	.1	3.4	.5	1.3	95
Corn, green	52	76.1	2.8	1.2	19.0	.8	.9	455
Peas, green	88	85.3	3.6	.2	9.8	1.2	1.1	255
Pumpkin	7	91.6	.8	.2	6.7	1.1	.7	150
Squash	5	87.6	.9	.5	10.5	.7	.5	235
Succotash	12	75.9	3.6	1.0	18.6	.9	.9	455
Tomatoes	19	94.0	1.2	.2	4.0	.5	.6	105
CANNED FRUITS.								
Apples, crab	1	42.4	.3	2.4	54.4	.....	.5	1,120
Apple sauce	1	61.1	.2	.8	37.2	.....	.7	730
Apricots	1	81.4	.9	.....	17.3	.....	.4	340
Blackberries	1	40.0	.8	2.1	56.4	.....	.7	1,150
Blueberries	3	85.6	.6	.6	12.8	.....	.4	275
Cherries	1	77.2	1.1	.1	21.1	.....	.5	415
Peaches	3	88.1	.7	.1	10.8	.....	.3	220
Pears	4	81.1	.3	.3	18.0	.....	.3	355
Pineapples	1	61.8	.4	.7	36.4	.....	.7	715
Strawberries	1	74.8	.7	.....	24.0	.....	.5	460

* U. S. Dept. of Agric., Exp. Sta. Bul. 28, p. 70.

Methods of Proximate Analysis.—As a rule, the contents of canned goods are intended to be entirely edible throughout, and contain little or no refuse or portions to be rejected. An exception to this is the occasional canning of certain fruits with stones or pits, which are, of course, to be removed. The can or package is first weighed before opening, and later the cleaned receptacle is weighed after its contents have been removed. The weight of the contents is thus ascertained by difference.

For the analytical determinations, the contents of the can or bottle are intimately mixed to form a homogeneous pulp, so that parts taken for analysis are fairly representative of the whole. If considerable liquid is present, with some solid masses held in suspension therein, the liquid is best drained off, and the solid portions pulped separately in any convenient manner, as by the use of a mortar, or by means of a household

food-chopper. The whole is then thoroughly mingled together. If desired, the weight of the liquid and solid portions may be separately ascertained before mixing.

The analyst should use judgment and discrimination as to how various portions of the mass are to be best measured out for the determinations. Much depends on the consistency of the pulpy mass. It is often convenient to make a 20% solution or mixture of the material with water, using say 50 grams of the pulped sample in 250 cc. of water, such of the sample as is insoluble being disintegrated by shaking.

Methods for determining water, ether extract, crude fiber, protein, and ash do not differ materially from those employed in the case of corresponding fresh fruits and vegetables.

These determinations, in the case of canned products, while useful in showing their food value, give little information as to their adulteration by the substitution of foreign vegetable or fruit pulp.

ACCIDENTAL IMPURITIES.—Under this head are included (1) products of decomposition, due to the incomplete sterilization of the contents of the can, and (2) metallic salts due to the solvent action of the juices of the contents on the inner surface of the can, or of the vessel in which the product has been previously cooked.

Decomposition, and the Detection of Spoiled Cans.—In the case of canned vegetable products, decomposition rarely results in the formation of ptomaines even after the can has long been open, though these toxins are sometimes formed, in canned meat and fish. Decomposition is readily apparent after opening a can, from a cursory examination of its contents. The appearance, taste, and odor will not fail to indicate the unfitness of the contents for food, if decomposition is at all advanced. It is, how-



of the cans is tested by sounding or striking the cans. If the contents are sweet, a peculiar note is produced when the can is struck, readily distinguishable from the dull tone of the unsound can by any one familiar with the work.

As stated above, concavity in the ends of the can indicates that the contents are in good condition.

Prescott and Underwood further state that sound cans may be distinguished from unsound in a lot of suspicious goods, when the swelling of the ends is not apparent, by the following method:

Boil the cans for an hour, causing the ends of all to swell, then cool, and set aside for eight hours, during which the sound cans will snap back, while the unsound will continue convex, by reason of the fact that the swelling in this case is due to the generation of gas by the bacteria present.

Examination of Gases from Spoiled Cans.—When the tops of blown cans are punctured in the process of opening, an outflow of gas is usually to be noted. Doremus * has studied the character of these gases and

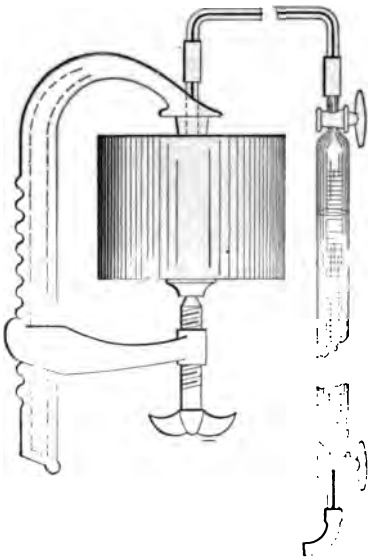


FIG. 118.—Apparatus for Collecting Gases from Spoiled Cans. (After Doremus.)

found that when the contents have become putrid, carbon dioxide and hydrogen are the chief gases to be found. Often 60 to 80 cc. of gas may be collected from a can. For the collection of the gases, Doremus

* Jour. Am. Chem. Soc., 19, 1897, p. 733.

uses the device shown in Fig. 118. An adjustable clamp has attached to its upper arm a beveled, hollow, steel needle. A perforated rubber stopper covers the needle and serves as a cushion. A fine tube connects the needle with the receiver of a eudiometer, both tube and receiver being filled with water or mercury. Either the stop-cock form of eudiometer, as here shown, or the kind with attached leveling-tube may be used. The can is adjusted between the arms of the clamp, and by turning the screw the needle is brought into contact with the top of the can and caused to puncture it, the rubber stopper serving to make a gas-tight joint. The gas passes through the tube into the eudiometer, and its constituents are determined in the usual manner, either by introducing the reagents directly into the eudiometer-tube in the proper order, or by transferring the gases to pipettes.* Hydrogen sulphide is tested for by subjecting a filter-paper moistened with lead acetate solution to the gas. If it turns black, the presence of hydrogen sulphide is indicated.

METALLIC IMPURITIES.

Salts of Lead and Tin are commonly met with in varying amounts in nearly all classes of products put up in tin. The quantity dissolved depends largely on the character of the tin plate used in the manufacture of the can, as well as on how the solder is applied. Much depends also on the nature of the food product and its acidity. Formerly much danger was apprehended from the use of the so-called terne plate as a material for cans. This consists of an alloy of lead and tin, coated on iron plate and intended for use as roofing. Sometimes two parts of lead to one part of tin are found in terne plate. Only the better



in which the contents have access to the solder, and it is common to find lumps of solder in the can from the final sealing of the tapped hole in the top. The amount of lead in 24 samples of solder taken from the interior of some of the cans mentioned in the preceding paragraph, was found to vary between the limits of 51 and 65 per cent.*

Action of Fruits and Vegetables on Tin Plate.—The amount of tin dissolved by various canned fruits and vegetables is roughly indicated by the corrosion of the inner surface of the can. A large variety of these canned products have been examined in the laboratory of the Massachu-

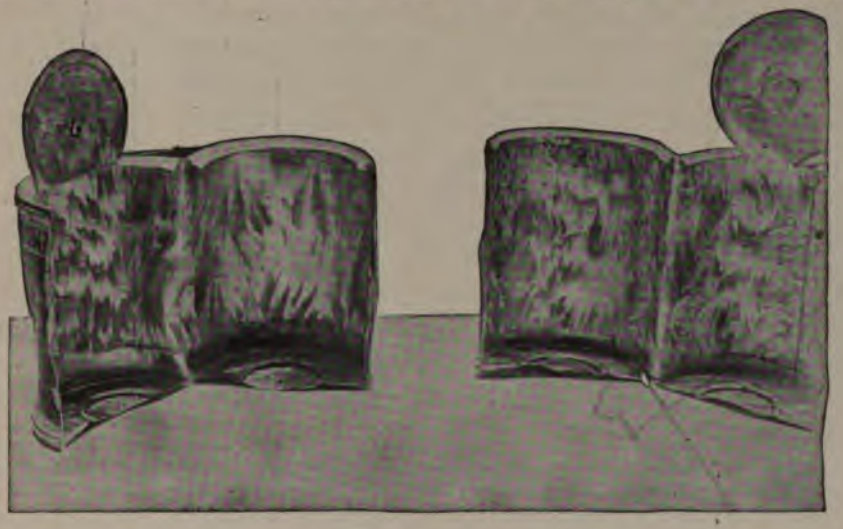


FIG. 119.—Interior of Blueberry Cans, Cut Open to Show the Corrosion by Acid of the Fruit Juice.

setts State Board of Health, with a view to determining the quantity of tin contained in solution. The results have shown that though notable traces of tin were found in acid fruits and rhubarb, and large traces in some green vegetables, canned blueberries were found to contain, as a rule, much more tin in solution than any other canned goods examined. It is assumed that the tin was, at least in considerable part, still held in solution by the fruit acids, inasmuch as the metal was found in the filtered juice. In every instance the inner tin lining was found to be extensively corroded, and in some cases it had been almost entirely dissolved off, leaving the underlying iron bare. Fig. 119 shows the appear-

* Bul. 13, p. 1038.

ance of one of these cans, split open to show its inner surfaces. The corrosion is apparent. Eleven samples of canned blueberries, representing seven brands, were examined in 1894 by Worcester, showing an amount of tin in solution (calculated as SnO_2) varying from 0.066 to 0.27 gram per can of 615 cc. capacity.

In 1899 samples of various canned products were examined for lead and tin in the author's laboratory, the results of which are thus summarized:*

	Tin, Grams.	Lead, Grams.	Capacity of Can, cc.
Strawberries	.....	.....	615
Highest	.0393	.0004	
Lowest	.0124	.0000	
Raspberries	.....	.....	615
Highest	.0848	.0002	
Lowest	.0725	.0001	
Blueberries	.....	.....	615
Highest	.2226	.0021	
Lowest	.0056	.0004	
Tomatoes	.....	.....	950
Highest	.0515	.0004	
Lowest	.0146	.0001	
String beans	.....	.....	650
Highest	.0499	.0003	
Lowest	.0065	.0008	
Peas	.....	.....	615
Highest	.0046	.0000	
Lowest	.0024	.0001	
Corn	.....	.....	615
Highest	.0101	.0011	
Lowest	.0045	.0001	
Lima beans	.0064	.0004	650
Succotash	.0039	.0001	650
Squash	.....	.....	950
Highest	.1793	.0087	
Lowest	.1577	.0003	
Pumpkin	.1844	.0010	950

also been examined by the Massachusetts Board, and found to be high in tin. The highest figures showed 0.376 gram (expressed as metallic tin) in a half-pound can. In these cases the corrosion of the interior of the cans was very marked.

Effect of Time on Amount of Tin Dissolved.—A series of experiments was conducted by the author in 1899 * on the action of various fruit acids on tin, with a view to ascertaining, among other facts, whether or not the element of time exerts an appreciable difference in the results.

Samples of various canned fruits and vegetables were titrated for their acidity. It was found that certain samples of canned blueberries, for instance, had an acidity of about one-twentieth normal. In the case of strawberries, the acidity was about one-sixth normal. Canned raspberries were found to be about one-tenth normal in acidity, while the acidity of canned tomatoes varied from one-tenth to one-fourteenth normal. Solutions of one-fifth, one-tenth, and one-fifteenth-normal malic acid, one-tenth and one-fifteenth-normal tartaric acid, one-tenth and one-fifteenth-normal citric acid, and one-tenth-normal acetic acid were prepared and sealed in pint glass jars, having about the same capacity as the ordinary-sized tin fruit cans, each jar containing an amount of tin plate equivalent to the interior exposed surface of a can. Solutions thus sealed were kept for three months, six months, and a year, and examined at the end of these respective periods for tin. The results showing the amount of tin found at the end of three months in each case are given in the following list:

ACTION OF FRUIT ACIDS ON TIN IN THREE MONTHS.

Acid.	Grams of Tin in One Pint of Solution.	Acid.	Grams of Tin in One Pint of Solution.
N/5 malic	0.0578	N/15 tartaric	0.0246
N/10 "	0.0201	N/10 citric	0.0374
N/15 "	0.0107	N/15 "	0.0236
N/10 tartaric	0.0382	N/10 acetic	0.0010

It was found, in general, that the amount of tin dissolved in three months, as indicated above, was the maximum amount dissolved, or, in other words, with a very few exceptions no additional tin was dissolved by added exposure to the acid for six months, or even a year. The

* An. Rep. Mass. State Board of Health, 1899, p. 624.

amount of tin dissolved was found to vary proportionally with the strength of acid, as would naturally be expected.

Experiments with tenth-normal acetic acid (which was found to be the approximate acidity of the canned sardines mentioned on page 697), sealed in jars with tin plate, as in the case of the fruit acids, and kept for three and six months respectively, showed that in three months 0.0019 gram, and in six months 0.0083 gram of metallic tin had been dissolved, indicating much less vigorous action than that of the same strength of fruit acids, and dissolving less tin than the samples of sardines examined.

Salts of Lead.—While it is a fact that the material of the tin plating usually found in cans is comparatively low in lead, the same is not always true of the metal caps used to cover some of the bottled goods. The French "haricots verts" are usually sold in wide-mouthed bottles, closed by a disk of very soft metal. In one instance this metal cap, which came in contact with the liquid contents of the bottle, was found to contain 93½% of lead. Of the various kinds of bottles in which are sold cheap carbonated drinks known as "pop," one style has a stopper consisting of a metallic button surrounded by a rubber ring. These metallic buttons consist of tin and lead in varying proportions. Inasmuch as the inclosed liquor was usually found to be quite acid in reaction, the danger of prolonged contact with the metallic portion of the stopper is evident.

The following table gives the percentage of lead found in the stoppers of this character, together with the amount of lead contained in the liquor:*

Character of Sample.	Per Cent of Lead in Stopper.	Amount of Lead in Contents of Bottle in Milligrams.†
Blood orange	59.7	0.31

contents of the bottles was in no case very large, it was enough to condemn the use of lead in the manufacture of such stoppers. That the amounts of lead found in the contents of the bottles vary quite irrespective of the percentage of lead in their stoppers, may be ascribed to various causes, such as the difference in the acidity of the liquors, and the length of time that the liquor has been in contact with the stopper. Furthermore, the more soluble metal of an alloy is attacked by an acid with an energy which is not proportional to the percentage of that metal in the alloy.

Salts of Zinc.—The presence of zinc salts in canned foods is largely accidental, and is generally due either to the contact of the acid fruits and vegetables with galvanized iron in the canneries, to the occasional use of brass vessels, or to the zinc chloride used as a soldering fluid. Hilgard and Colby * have examined empty tin cans fresh from the manufacturer, and found zinc chloride in notable quantity in the seams, and especially in the empty space of the lap at the bottom of the can, where it could easily be acted on by the contents. The average amount of soluble zinc chloride found in the "lap" alone amounted to three-fourths of a grain per can. It was furthermore ascertained that it was not the practice of canners to wash the cans before packing, so that zinc present in canned goods may thus readily be accounted for.

Zinc chloride is commonly used in machine soldering, but should be displaced by rosin.

Hilgard and Colby found in some spoiled cans of asparagus, where the acidity was unusually high, an average of 6.3 grains of zinc chloride per large can.

Zinc salts are said to have been used for greening peas, but their use for this purpose is not common. Zinc chloride is the salt used, and a natural yellowish-green tint is imparted when properly applied. The process has been kept secret.

Salts of Copper.—While copper in canned goods is sometimes accidental, its presence being due to the use of copper or brass vessels in the canneries, its chief interest to the food analyst lies in the use of copper sulphate for greening peas and other vegetables. The artificial greening of vegetables is much more commonly practiced in France than in the United States.

French canners of peas, beans, Brussels sprouts, etc., are frequently so lavish in the use of sulphate of copper that the goods as found on our

* Rep. Cal. Agric. Exp. Sta., 1897–8, p. 159.

markets can in some cases hardly be said to resemble the freshly cooked products in color. Oftentimes, indeed, they possess such a deep green as to be positively distasteful to the average American palate, though evidently this unnatural hue is craved in Europe. The use of copper in such foods is often rendered apparent by the most cursory examination.

In this country, when copper is used, smaller quantities are usually employed, with an attempt to imitate more closely the color of the natural product.

Complaint in court for this form of adulteration under the general food law as it exists in most states would naturally be brought under one of two clauses:

1st. As being colored, whereby the product appears of greater value than it really is, or

2d. As containing an ingredient injurious to health.

An ingenious claim is sometimes advanced by the defendant in opposition to clause 1, to the effect that copper sulphate is added, not to give an artificial green color, but to preserve the original green of the chlorophyl or natural color of the fresh peas,* so that it will not be destroyed by subsequent boiling.

This point was argued in a strongly contested court case brought in Massachusetts for copper in French peas.†

As Worcester ‡ has shown, the fallacy of this argument can be easily demonstrated. If it were true that the copper acts as a preservative of the chlorophyl, a pure extract of chlorophyl should, by the addition of copper sulphate, be prevented from destruction on boiling, and again, on once destroying the color of the chlorophyl by boiling, it would be impossible to restore it by further boiling it with copper sulphate.



by boiling albumins with copper salts, due to the formation of albuminate, or in the case of peas, leguminate of copper. Harrington * states that it is possible to color eggs an intense green by boiling with copper sulphate.

Examination of a large number of brands of canned vegetables greened by copper, as bought in Massachusetts, showed that the amount used varied from a trace to 2.75 grams per can, calculated as copper sulphate. In justice to the consumer, who may be cautious about taking into his system copper salts, as well as to those who are indifferent to their use, it is no more than fair that all cans should have a label, plainly stating the quantity present. In the Massachusetts market, labels like the following are not uncommon: "This package of French Vegetables contains an equivalent of Metallic Copper not exceeding three-quarters of a grain."

Copper as a coloring matter has been most commonly found in peas, beans, and Brussels sprouts. Copper salts in minute quantity have been found in Massachusetts in canned tomatoes, clams, and squash, as well as in pickles.

Salts of Nickel.—Sulphate of nickel has been employed instead of sulphate of copper for greening vegetables. According to Harrington † 0.25 gram of nickelous sulphate per kilogram of peas is used. The peas or other vegetables are boiled in a solution of the salt, made slightly alkaline with ammonia.

Toxic Effects of Metallic Salts.—Divergence of opinion is so great as to the toxic effects of salts of the heavy metals on the human system, when present in the small amounts commonly found in food products, that it is extremely difficult to maintain a complaint in court based entirely on the harmful effects of these salts. Since the question is one for the toxicologist or physiological chemist rather than the analyst to settle, it will not be discussed here at length; suffice it to say that a large number of experiments on human beings will undoubtedly have to be tried, before the necessary data will be at hand on which to base a really intelligent opinion. The same general difficulties are met with here as one encounters in the matter of determining the definite effects of antiseptics in food, of alum in baking-powder, etc.

Determination of Lead in Tin Alloy.—*Method of Paris Municipal Laboratory.*‡ The material, if soft, is hammered into a thin plate, and

* Practical Hygiene, p. 203.

† *Ibid.*, p. 205.

‡ Analyse des Matières Alimentaires et Recherche de leurs Falsifications, 1894, p. 695.

2½ grams are weighed out, transferred to a 250-cc. flask, and dissolved in 7 to 8 cc. of concentrated nitric acid. Evaporate to dryness on the sand-bath, add 10 drops of nitric acid and 50 cc. of boiling water, cool, and make up to 250 cc. with water. Let the residue settle and pour off through a filter 100 cc. of the clear, supernatant liquid, corresponding to 1 gram of the material. This contains the lead, while the tin is left behind in the residue, together with antimony if present.

Add 10 cc. of a standard solution of potassium bichromate (7.13 grams to the liter) and shake. Each cubic centimeter of this standard solution is sufficient to precipitate 0.01 gram of lead. Allow the lead chromate formed to settle, and, if the solution is colorless, add 10 cc. more of the bichromate, or sufficient to be present in excess, as indicated by the yellow color. Filter, wash, and titrate the excess of bichromate with a standard iron solution, containing 57 grams of the double sulphate of iron and ammonia and 125 grams of sulphuric acid per liter. This iron solution should be kept under a layer of petroleum, and standardized against the potassium bichromate before use.

Add, drop by drop, the iron solution to that containing the excess of bichromate. The color of the latter passes from pale green to bright green, when the chromate is completely reduced. Determine the end-point with a freshly prepared dilute solution of potassium ferricyanide, a drop of which is placed on a porcelain plate or tile in contact with a little of the solution titrated. A blue color is produced when the iron is present in excess. If the standard iron and bichromate solutions exactly correspond, 1 cc. of the iron solution is equivalent to 1% of lead, but the latter solution is usually a little weak.

If n = number of cubic centimeters of iron solution necessary to reduce 10 cc. of the standard bichromate



first evaporated to dryness, and from 10 to 15 cc. of concentrated sulphuric acid or enough to carbonize are added to the dry residue contained in a porcelain evaporating-dish, which is very gently heated over the flame till foaming ceases. Then ignite to an ash in a muffle, or carefully over the free flame, using a little nitric acid, if necessary, for oxidation of the organic matter. Add 20 cc. of dilute hydrochloric acid, and evaporate over the water-bath to dryness. Wash the residue into a beaker, slightly acidify with hydrochloric acid, and saturate with hydrogen sulphide without previous filtration. Heat the beaker on the water-bath, and pass the contents through a filter. Wash the precipitate, which contains sulphides of tin, lead, and copper, if these metals are present, while if there is zinc, it is contained in the filtrate. The precipitate is fused with sodium hydroxide in a silver crucible for half an hour, to increase the solubility of the tin, which would otherwise be dissolved with difficulty. The fusion is boiled up with hot water, acidulated with hydrochloric acid, and transferred without filtering to a beaker, in which hydrogen sulphide is added to saturation. This precipitates the sulphides of tin, lead, and copper (if these metals are present). The sulphide precipitate is collected on a filter, and thoroughly washed with hot water, the washings being rejected. Pass through the filter several portions of boiling ammonium sulphide, using about 50 cc. in all, or till all the tin is dissolved. Precipitate the tin from the combined filtrate with hydrochloric acid, filter, wash, ignite, and weigh as stannic oxide.

The residue left on the filter, after dissolving out the tin sulphide, is then dissolved by treatment with nitric acid, which is filtered, and to the filtrate and washings ammonia is added nearly to the point of neutralization. Then add ammonium acetate. Filter off any precipitate of iron that may be formed. The filtrate is divided into two portions for determination of copper and lead. If lead is absent, determine the copper by titration with potassium cyanide * or electrolytically (p. 403). Copper is rarely present in sufficient amount to be determined, unless used for greening the vegetables. If notable quantities of lead are present, the solution is made acid with acetic, and the lead precipitated therefrom with potassium chromate, collected on a tared filter, washed with water acidified with acetic acid, dried at 100° C., and weighed as lead chromate. Or determine the lead by color-tests, as on page 704.

For the determination of zinc, the filtrate from the first hydrogen-sulphide residue is evaporated to a volume of about 60 cc., and treated

* Sutton, Volumetric Analysis, 8th ed., p. 204.

with bromine water to oxidize the iron, as well as any excess of hydrogen sulphide remaining, the excess of bromine is then boiled off, and a few drops of concentrated ferric chloride added, to make the solution distinctly yellow, if not already so. Nearly neutralize with ammonia, and precipitate the iron with ammonium acetate. Filter, wash, acidify the filtrate with acetic acid, and precipitate the zinc with hydrogen sulphide. Filter, wash, ignite, and weigh as zinc oxide.

The metals may be determined separately, as follows:

Determination of Tin.*—Evaporate the contents of the can to dryness, and ignite in porcelain. Fuse the ash with sodium hydroxide in a silver crucible, boil the fusion with several portions of water acidulated with hydrochloric acid, filter, and precipitate the tin from the acid solution with hydrogen sulphide. Dissolve the washed precipitate in ammonium sulphide, filter, and deposit the tin directly from this solution by electrolysis in the platinum dish which contains it, using a current of 0.05 ampere and the electrolytic apparatus described on page 493.

Determination of Lead, especially applicable if lead is present in small amounts only. Boil the sulphated ash of the contents of the can (obtained as on page 703) with a solution of ammonium acetate, having an excess of ammonia. The tin, zinc, and iron remain insoluble, while the copper and lead are dissolved. Filter, wash, and add a few drops of potassium cyanide to the filtrate, to prevent precipitation of copper when hydrogen sulphide is subsequently added. If the solution exceeds 40 cc., concentrate to that amount by evaporation, and transfer to a 50-cc. Nessler tube. Add hydrogen sulphide water, and make up to the mark. Compare the brown color imparted by the lead sulphide, with the colors obtained by treating with hydrogen sulphide water in Nessler tubes



(2) *Colorimetrically.*—This method is especially applicable for small amounts of copper. The blue-colored ammoniacal solution of the ash, filtered as in (1), is transferred to a Nessler tube, and its color matched against the colors of a series of measured amounts of an ammoniacal standard solution of copper sulphate

Determination of Nickel.—Boil the ash with water slightly acidified with hydrochloric acid, and without filtering, saturate with hydrogen sulphide, thus precipitating out any copper, tin, or lead. Filter and wash. Zinc and nickel, if present, are in the filtrate. Boil the filtrate to expel the hydrogen sulphide, and add sodium carbonate till slightly alkaline. Add acetic acid without filtering till the precipitate produced by the alkaline carbonate is dissolved, and then add a considerable excess of acetic acid. The zinc is precipitated by passing hydrogen sulphide through the cold dilute solution, while the nickel is held in solution by the large excess of acetic acid. Filter, and wash with hydrogen sulphide water, to which a little ammonium acetate has been added.

Make the filtrate alkaline with ammonia, precipitate the nickel with ammonium sulphide, filter, wash, ignite, and weigh as nickelous oxide.

ANTISEPTICS IN CANNED FOODS.

No class of food products stands so little in need of these added substances to arrest fermentation as canned foods, if properly prepared; but, as a matter of fact, the use of antiseptics in this connection is on the increase. Prolonged heating for a sufficient length of time to perfectly sterilize the contents of a can is in some cases more or less detrimental to the appearance of the product, so that for this reason, as well as to save time in "processing," and furthermore to increase the keeping qualities of the goods after opening, many manufacturers resort to the use of artificial chemical preservatives. So long as any well-founded prejudice against preservatives exists, their use in canned or bottled foods should be unequivocally condemned, unless the cans or packages are distinctly labeled with the nature of the preservative and the extent to which it is employed.

As in other foods, discrimination as to locality is apparently used on the part of manufacturers in shipping canned goods containing added preservatives, so that, as a matter of fact, in states where it is well understood that a vigilant enforcement of the pure-food law prevails, we do not find as high a percentage of canned foods with preservatives as in other states.

Preservatives commonly employed in canned goods are salicylic, benzoic, and sulphurous acids, though the other familiar antiseptic agents may be used. In such foods as canned corn, while the purpose of sulphurous acid may be in part as a preservative, the primary object for its use is undoubtedly to bleach or whiten the product.

The Bleaching of Corn by artificial means before canning is usually accomplished by boiling the corn with sulphite of soda, thus giving to the product an unnaturally white color. The practice seems to have been more in vogue ten years ago than at present, the popular taste now apparently preferring the natural rich yellow of fresh corn.

Saccharin is claimed to possess antiseptic powers and is used in canned goods, but its primary purpose is as a sweetener.

Beta-naphthol is also said to be used as a preservative in canned goods, but has not been found by the author in any samples that have come to him for analysis.

Detection of Preservatives.—Tests for salicylic or benzoic acid are most readily made in the residue from an ether extract of a portion of the acidified contents of the can or package, while formaldehyde and sulphurous acid are tested for in the earlier portions of the distillate, obtained by distilling a mixture of the acidified contents in water.

If it is desired to systematically test for the various preservatives in the same sample, a convenient method of procedure is as follows:

Thoroughly mix 50 grams of the pulped sample with water in a 250-cc. graduated flask, make distinctly acid with dilute phosphoric acid, and fill to the mark with water. Transfer to a distilling-flask, and subject to distillation in a glycerin- or paraffin-bath, whereby the temperature is raised near the end of the distillation considerably above 100° C.

The residue left in the flask is then washed out and incinerated, and the ash examined for boric acid.

"SOAKED GOODS."

It has become quite common, especially in the case of peas, beans, and corn, to utilize for canning purposes those that have grown old and dried, after soaking them for a long time. The presence of soaked peas in the market is generally more common in years when there is a scarcity in the pea crop. By the process of soaking, dried and matured field corn may be softened to such an extent as to be substituted for green or sweet corn in the canned product. These goods, frequently sold at a very low price, under some such tempting name as "Choice Early June Peas," are entirely devoid of that succulent property so highly prized in the fresh goods, and are altogether so inferior in quality that their sale may justly be considered as fraudulent, unless their character is specified. In some states the law provides that such a product, to be legally sold, shall have plainly marked on the label of the can the words "Soaked Goods" in letters of prescribed size.

Detection.—Methods of detecting soaked goods are distinctly physical rather than chemical. The appearance and taste of the goods furnish in most cases an unmistakable clue to their nature. Soaked goods are entirely lacking in juiciness, and in the flavors so characteristic of the various vegetables, when gathered and canned before becoming dry. The process of soaking is also said to develop the growth of the rudimentary stem of the embryo in the dried pea and bean. Peas and beans of the soaked variety are almost entirely lacking in the green color of the fresh vegetables, unless the color has been artificially supplied.

In all cases it will be found that the solid grains or kernels of the peas, beans, and corn that have once been dried, though softened by the process of soaking, have much less water than the grains of the corresponding vegetables that were gathered while still soft and succulent.

KETCHUPS AND TABLE SAUCES.

These preparations vary widely in their character and composition, and, in the absence of standards fixed by law for each particular mixture, almost any food product may be included in its make-up, without laying it open to the charge of being adulterated. At the same time, in this class of condiments it is naturally expected that the ingredients used all have some food value, and in addition possess a certain degree of pungency

or distinctive flavor. In other words, inert materials used simply as

CHEMICAL COMPOSITION OF KETCHUP, PICKLES, AND RELISHES.*

	Number of Analyses	Refuse.	Water.	Protein.	Fat.	Total Carbo-hydrates	Ash.	Fuel Value per Pound.
Tomato ketchup.....	2	.....	82.8	1.5	.2	12.3	3.2	265
Horseradish.....	2	.....	86.4	1.4	.2	10.5	1.5	230
Olives, green:								
Edible portion.....	1	.....	58.0	1.1	27.6	11.6	1.7	1,400
As purchased.....	1	27.0	42.3	.8	20.2	8.5	1.2	1,025
Olives, ripe:								
Edible portion.....	1	.....	64.7	1.7	25.9	4.3	3.4	1,205
As purchased	1	19.0	52.4	1.4	21.0	3.5	2.7	975
Cucumber pickles	3	.....	92.9	.5	.3	2.7	3.6	70
Mixed pickles.	1	.....	93.8	1.1	.4	4.0	.7	110
Spiced pickles.	1	.....	77.1	.4	.1	20.7	1.7	395

* U. S. Dept. of Agric., Office of Exp. Sta., Bul. 28, p. 70.

"fillers" are, to say the least, out of place, even though they are not actually adulterants.

Tomato Ketchup in its simplest form consists of boiled, fresh, ripe tomato pulp mixed with various spices, either with or without the addition of red peppers, sugar, and vinegar. The preparation, being usually strained through a sieve before bottling, is of smooth consistency and free from masses of pulp.

The ketchup of the housewife is made from varying recipes, all based on the above method of procedure; and while the commercial bottled ketchups should be made from materials quite as pure, it is often true, especially in the cheaper varieties, that the skins and refuse of tomato-canning factories form the basis of much of the ketchup in the market. Even with the use of these materials, when properly prepared, and before advanced fermentation has set in, with clean methods of handling, the



Coloring of Tomato Ketchup.—The practice of adding artificial dyestuffs to ketchup is very prevalent, but the brilliant magenta and crimson hues often imparted to the bottled ketchups on the market in no wise resemble the natural dull-red or brown color of the pure home-made article, in which the bright color of the fruit pulp is modified by the mixture of spices with which it is cooked. It is doubtless true that many manufacturers employ such inferior materials that unless some dyestuffs were added the result would be most unappetizing in appearance. Use of artificial coloring matters is not, however, universal. Out of ninety-five samples of tomato ketchup examined in 1901 in Connecticut, only fifteen were found uncolored.*

Anilin dyes and cochineal are most commonly used for coloring.

Walnut Ketchup.—This is made up in a somewhat similar manner to tomato ketchup, excepting that instead of tomatoes, soft young walnuts are crushed and used as a basis.

Chili Sauce is made up of a pulped mixture of tomatoes, red peppers, onions, vinegar, and various spices, differing from ketchup in that it contains the seeds and is not strained. In consistency it is heavier than ketchup. It is colored and preserved in much the same manner as ketchup.

Table Sauces are composed of a large variety of materials of a more or less pronounced flavor or pungency, combined in a liquid preparation usually of a more thin or watery consistency than the ketchups. The materials employed include mushrooms, onions, garlic, ground anchovies, tamarinds, spices, coriander and cardamom seeds, walnuts, vinegar, molasses, and even assafœtida. These bottled preparations are very rarely colored except with caramel, but sometimes contain antiseptics, especially salicylic and benzoic acids.

The Acidity of ketchups and table sauces furnishes a ready means of comparison between different varieties, and is conveniently expressed in terms of acetic acid.

To determine the acidity, titrate 1 gram of the diluted sample with tenth-normal sodium hydroxide, using phenolphthalein as an indicator.

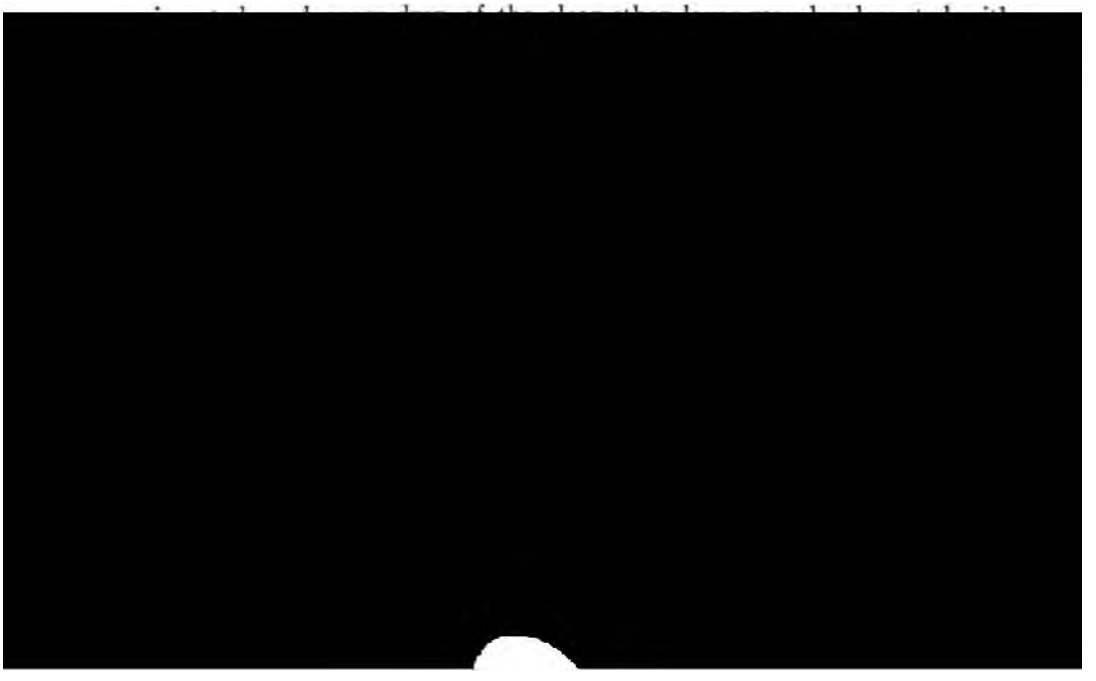
"225 bbls. new goods, made from waste; has *benzoate of soda* in it, packed in uncharred whiskey and wine barrels at $3.00 per bbl. net cash." "300 bbls. old goods, partly whole stock, partly waste, *has salicylic acid in it;* nice goods, etc. Price $2.60 per bbl." "400 bbls. new goods, Jersey style; solid and *good red color*, fine quality. Price $3.00 per bbl." With prices as low as the above quotations, it is difficult to see how a cheaper basis for ketchup stock than the above could be supplied. Even the pulp of pumpkin and of other inert vegetables, alleged to be used as adulterants, would hardly be furnished so cheaply.

* An. Rep. Conn. Exp. Sta., 1901.

Each cubic centimeter of the alkali corresponds to 0.006 gram of acetic acid.

Winton and Ogden * have found the acidity of tomato ketchups examined by them to vary between the limits of 0.60 and 2.20 per cent, calculated as acetic acid, Chili sauce from 0.80 to 1.80 per cent, and various table sauces from 1.40 to 1.60 per cent.

Examination of Table Sauces and Ketchups for Preservatives.— Extract a portion of the acid sample with ether or chloroform, which removes salicylic or benzoic acid or saccharin. If the sample is of thin or watery consistency, like most table sauces, the extraction can in most cases be readily effected in a separatory funnel, chloroform being in this case most convenient, since it sinks to the bottom. If ketchup or other thick syrupy substance is to be examined, it is almost impossible when shaking with ether or chloroform to avoid the formation of an annoying emulsion, which it is difficult to break up. For this reason the author prefers, in the case of ketchups and similar viscous fluids, to separate the extract by means of a centrifuge of the style shown in Fig. 11. A portion of the acid sample, say 75 cc., is shaken violently in a corked flask with 25 to 40 cc. of ether, and the mixture, usually in the form of an emulsion, is poured into two of the centrifuge tubes, so that they contain equal amounts and balance each other. They are then corked, placed in the shields of the centrifuge, and whirled from two to four minutes, or until the emulsion is broken up. At the end of this time it is usually found that the mixture is separated into three layers: first a watery layer at the bottom of the tube, then an almost solid layer of the viscous material above it, and finally the clear ether extract at the top. As a rule the separation is so complete that the tube may be



PICKLES.

A large variety of vegetables and fruits are preserved in the form of pickles in vinegar, either with or without spices, and kept in wooden pails, stoneware pots, kegs, or sealed, wide-mouthed bottles. The containers are not of necessity air-tight. The commoner vegetables are usually pickled without cooking, while with fruits, as in the case of peaches, pears, gooseberries, etc., they are usually cooked, or at least heated.

Cucumber Pickles are the most common, and are prepared by soaking the fresh cucumbers in strong salt brine. They are then dried on frames, and afterwards treated with boiling vinegar, to which spices may or may not be added. Other vegetables pickled in similar manner, either separately or in mixture with cucumbers to form "mixed pickles" or "gherkins," are cauliflower, bean pods, white cabbage, young walnut, and onions.

Such soft vegetables as young podded beans and beets are not treated with brine, but, after soaking in water, are directly treated with vinegar. The vinegar used for the finest pickling is of the cider, wine or malt variety. Cheaper varieties of pickles are put up in "white wine" or spirit vinegar.

Mustard Pickles.—These differ from plain vinegar pickles in the character of the preserving medium, which in this case consists of a mixture of mustard and spices with the vinegar to form a thin paste.

Piccalilli consists of a mixture in vinegar of various chopped vegetables, such as cucumbers, cauliflower, green pickles, onions, green tomatoes, and various spices.

Olives or pickling are picked before they have fully ripened, and the inherent bitter taste is removed by soaking in a solution of potash and lime. This is replaced by cold water, and finally the olives are transferred to the medium in which they are bottled, which consists of salt brine, either with or without flavoring. The flavoring materials employed consist of such substances as fennel, coriander, laurel leaves, and occasionally vinegar.

Capers.—These are the flower buds of the shrub *Capparis spinosa*, which are pickled in vinegar. Nasturtium seeds, when similarly pickled, possess a flavor much resembling capers, but their substitution for capers could readily be detected by their distinctive appearance, even if colored.

Adulteration of Pickles.—Green pickles, such as cucumbers, are

not uncommonly colored artificially by copper salts, either through the addition of copper sulphate, as in the greening of peas, or by the use of copper vessels. This artificial greening is to be looked for also in such products as capers and olives.

For methods of detection and estimation of copper, see page 704. Pickles may be greened by boiling with much less harmful substances than copper salts, such, for example, as grape leaves, spinach, or parsley.

Free Sulphuric Acid has been found in a number of cases in the vinegar of pickles bought on the Massachusetts market. A pronounced test for chloride with nitrate of silver should not be attributed to free hydrochloric acid, since it may be and probably is due to the salt from the brine in which the pickles have been treated.

Alum is sometimes added to the salt solution to produce hardness and crispness in pickles. A number of samples of cucumber pickles have been found by the author to contain alum. For its detection, fuse the ash of the pickles, if free from copper, in a platinum dish with sodium carbonate, extract with boiling water, filter, and add ammonium chloride. A flocculent precipitate shows alum.

Horseradish.—This condiment is prepared by grating the root of the perennial herb *Nasturtium armoracia*, and preserving in vinegar. It is very pungent and aromatic when first prepared, but by exposure to light and air quickly loses strength. A common adulterant of bottled horseradish as sold on the market is the ordinary grated turnip, the presence of which can be readily detected by means of the microscope.

JAMS AND JELLIES.

Jams or marmalades are prepared from the pulp of fruits, and jellies

till soft, transferred to a bag or press, and the juice allowed to flow out spontaneously, or is squeezed out under pressure, according to the grade of jelly desired, the clearest and finest varieties being made from the juice that flows out naturally. This juice is then evaporated down with the addition of sugar to a density of from 30° to 32° Bé., which is of the proper consistency to form a perfect jelly product after cooling, and, while still hot, is poured into the tumblers in which it is to be kept. Here, as in the case of jams, the amount of sugar varies, some using pound for pound, and others only half as much sugar as fruit. Some manufacturers clarify their jellies by mixing with the juice, while boiling, elutriated chalk, using a teaspoonful to each quart of juice. The impurities come to the surface with the chalk as a scum, and are skimmed off. This clarifying process is somewhat analogous to the defecation of sugar juices with lime, and is commonly carried out with apple jelly.

The "jellying" or gelatinizing of the final product is due to the presence in the fruit juice of pectin, or so-called vegetable jelly ($C_{32}H_{40}O_{28}4H_2O$); see page 217.

The high content of added sugar in jelly, once thought to be essential for keeping it, is now no longer considered necessary, and much less sugar is at present added than formerly. The finest grade of apple jelly, for instance, is made without any added sugar whatever.

In making the better grades of apple jelly, apple juice fresh from the press is run directly into the boiler or evaporator before any fermentation has ensued, and gelatinized by concentration. If boiled cider is wanted instead of jelly, it is drawn off at an earlier stage than in the case of apple jelly.

Composition of Known-purity Jellies and Jams.—In the tables on pp. 714 and 715, due to Tolman, Munson, and Bigelow,* are given results reached on the examination of the pure finished products, as well as on pure fruit juices and pulp used in their manufacture.

Adulteration of Jams and Jellies.—As a matter of fact, a small percentage of these products sold in the United States are honest prototypes of the home-made jams and jellies, which consist exclusively of the fruit specified on the label, in mixture with pure cane sugar. If we accept as a standard the product of the housewife, fully 90% of the commercial brands of these preparations would be found wanting. So great is the demand for cheap sweets of this variety, that the market is flooded with them at eight and ten cents per half-pound jar, when in reality abso-

* Jour. Am. Chem. Soc. (1901), pp. 349–351.

TABLE I. [illegible]

TABLE II. [illegible]

[redacted]

TABLE III.—FRUIT.*

Description of Sample.	Total Solids, Per Cent.	Ash, Per Cent.	Total Acids Calculated as H_2SO_4, Per Cent.	Proteids (N×6.25), Per Cent.	Sugars.				Polarization.		
					Reducing Sugars, Per Cent.	Cane Sugar Added, Per Cent.	Cane Sugar Found, Per Cent.	Added Cane Sugar Inverted, Per Cent.	Direct at 18° C.	Invert at 18° C.	Invert at 86° C.
Apple (fall pippin)	8.25	0.30	0.499	.....	4.13	.....	1.03	.....	−2.2	−3.6	−1.8
Blackberry	9.62	0.60	0.916	0.725	5.67	.....	.....	.....	−1.1	−1.0	0.0
Crab apple	14.34	0.84	0.705	0.418	5.68	.....	1.70	.....	−0.7	−3.0	−0.8
Grape (Ives seedling)	12.50	0.75	.....	.....	6.11	.....	0.29	.....	−1.6	−2.0	0.0
Orange (Florida navel)	13.11	0.61	0.686	0.985	4.13	.....	3.33	.....	+2.0	−2.5	−0.8
Pineapple	13.71	0.50	0.392	0.056	8.12	.....	3.11	.....	+1.0	−3.2	−0.0
TABLE IV.—JAM.											
Apple (fall pippin)	63.22	0.20	0.282	0.175	25.52	51.31	29.11	43.22	+26.3	−13.0	+4.8
Blackberry	55.42	0.48	0.851	0.737	18.77	43.99	29.00	34.08	+24.6	−14.6	+1.6
Crab apple	41.82	0.27	0.715	0.493	14.80	36.09	23.04	35.46	+18.3	−12.8	−0.6
Grape (fox)	61.80	0.19	0.698	0.200	50.06	54.21	3.70	92.96	− 9.0	−14.0	+2.2
Grape (Ives seedling)	56.64	0.48	0.744	0.525	33.44	42.45	11.33	73.38	+ 3.5	−11.8	0.0
Orange (Florida navel)	80.52	0.44	0.433	0.944	13.61	69.13	54.23	21.55	+55.9	−17.5	+2.0
Pear (Bartlett)	61.52	0.28	0.163	0.312	13.20	46.52	33.74	18.87	+32.3	−13.2	+1.0
Pineapple marmalade	73.92	0.30	0.315	0.312	14.05	60.20	46.40	22.90	+52.3	−10.3	+6.2
Plum (Damson)	50.43	0.54	1.012	0.525	28.29	37.75	9.70	74.42	+ 3.1	−10.0	+1.2
Plum (wild fox)	62.10	0.46	1.355	0.212	28.78	47.86	23.26	53.43	+13.9	−17.5	0.0

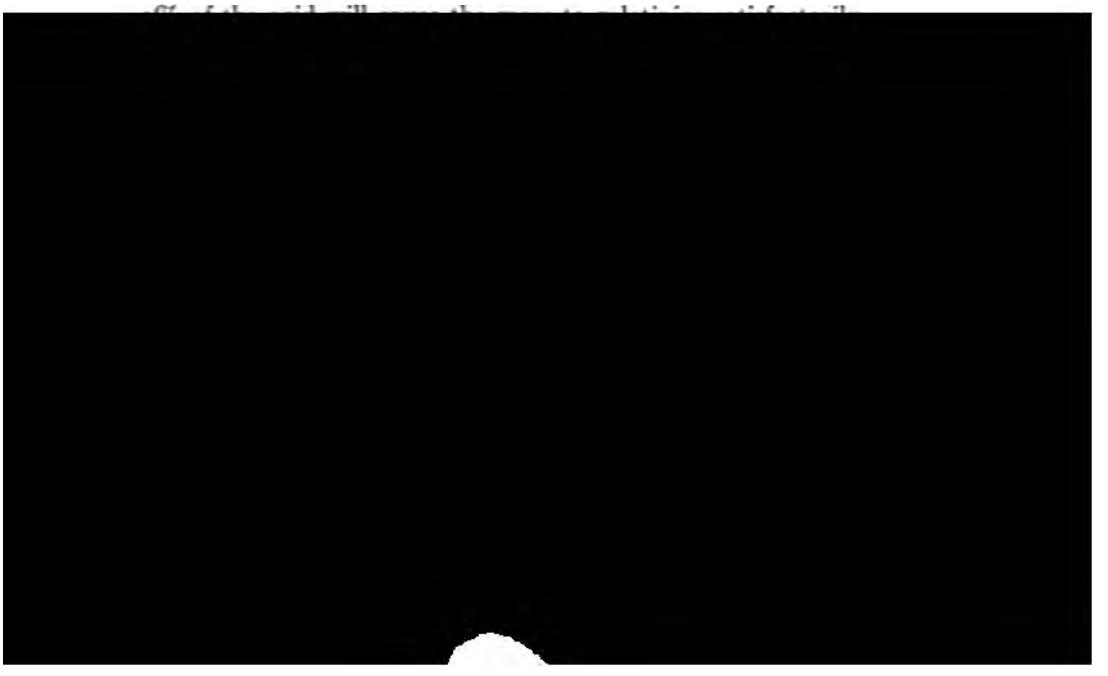
*The composition here given is not that of the original fruit, but of the pulped mass used in the preparation of jams.

lutely pure goods cannot be produced at much less than twice that amount.

The cheap substitutes are made up largely of apple juice and commercial glucose, frequently containing no fruit whatever of the kind specified on the label. Sometimes an attempt is made to imitate the flavor by the addition of artificial fruit essences, but more often the same apple-glucose stock mixture of jelly, put out under a particular brand, serves to masquerade as damson, strawberry, raspberry, current, grape, etc., differing from each other only in color, but not as a rule in flavor. A variety of artificial colors are employed, mostly coal-tar dyes. To compensate for the lack of sweetness of the glucose, a minute quantity of one of the concentrated sweeteners, such as saccharin or dulcin, is sometimes added. Besides artificial colors, antiseptic substances are commonly employed, especially salicylic and benzoic acids.

All grades of apple stock are found in these preparations. A large source of supply is furnished by the parings and cores of canning establishments, to say nothing of the refuse of these factories, such materials being boiled with water, and the extract, variously colored to imitate the different fruits, being evaporated with commercial glucose.

Adulterated Jelly.—While it is easy to make an excellent apple jelly by simple evaporation of the pure apple juice, even without the addition of sugar, it is impossible, or at least difficult, to obtain the proper degree of stiffness with a mixture of apple stock and commercial glucose. It is customary, in the manufacture of cheap jellies, therefore, to employ what is technically termed a "coagulator," consisting commonly of sulphuric acid, to which sometimes alum is added. Citric or tartaric acid is also used for this purpose, as well as to increase the acidity. About



Adulterated Jams.—Most of the cheap jams and bottled preserves sold on the market, though reinforced with apple stock, do in reality contain masses of fruit and berries of the kind stipulated on the label, as even a casual megascopic examination will show. That such low-priced preparations really contain genuine fruit pulp is not to be wondered at, when it is considered that much of the virtue of this fruit has sometimes been previously extracted by boiling, to produce fruit juices for higher-priced goods. Or, as in the case of jams containing strawberries, raspberries, and other small fruits with seeds, the juice is apt to have been previously expressed for pure jellies, while the residues are afterwards worked up with apple stock for low-priced jams. Hence the presence of pure fruit stock, or genuine berry seeds and pulp in jams, is in itself no criterion of purity, and, furthermore, it is unnecessary to use hay seed and other alleged foreign seeds as adulterants of cheap jam.

Compound Goods.—Many states have a law legalizing the sale of "compound" goods, providing they are distinctly so labeled. In other states, as, for instance, Massachusetts, the label must plainly state the name and percentage of the ingredients. In either case the analyst must discriminate, in classifying the inferior or low-grade preparations, between those that are labeled in accordance with the law, and those that are not. Only those not properly labeled can in such cases be classed as adulterated within the meaning of the law. Where such a law prevails, probably no class of food-products is so extensively affected by it as the low-grade jams, preserves, and jellies.

The restrictions as to labeling do not in all cases eliminate the element of deception. It is hardly justifiable, for example, to boldly label an alleged "currant jelly" which contains no currant, in the following manner:

Fruit juice	25%
Cane sugar	14%
Corn syrup	61%
	100%

The use of the term "fruit juice" surely implies to the unsuspecting purchaser that so much pure currant juice has entered into the jelly, elsewhere labeled in large letters "Currant," whereas all the juice is apple, and no currant juice has been used.

The following label is a type of those which discriminate between pure fruit and apple juice:

Fruit	30%
Corn syrup	35%
Granulated sugar	15%
Apple juice	20%
	100%

Composition of Cheaper Grades.—Out of 66 samples of jellies, jams, and preserves analyzed by Winton, Langley, and Ogden in Connecticut, the samples being purchased in that state,* 17 samples contained starch paste, 35 were artificially colored with coal-tar dyes, and 19 contained salicylic or benzoic acid.

The following table has been compiled, showing the sugar content of some of the typical commercial jellies and jams analyzed in the laboratory of the Massachusetts State Board of Health. Nearly all of these were artificially colored, and found to contain little if any fruit, other than apple.

	Direct Polarization.	Invert Polarization.		Per Cent Sucrose.	Per Cent Commercial Glucose.
		At 20° C.	At 87° C.		
JELLY.					
Apple	+64.0	+28.0	+36.0	26.8	22.1
Currant A	+29.2	+20.0	+36.4	6.9	22.3
" B	+41.6	+33.9	+40.8	5.7	25.0
Grape	+62.0	+34.4	+46.0	20.6	28.2
Peach	+119.8	+108.8	+110.0	8.2	67.4
Pineapple	+114.0	+107.6	+110.0	4.9	67.4
Raspberry	+112.0	+92.0	+93.6	14.9	57.4
JAM.					
Damson A	+107.0	+94.4	+58.1	9.3	35.6
" B	+95.2	+90.9	+83.6	3.2	51.2
Apricot	+99.0	+93.5	+85.6	4.1	52.4
Quince	+49.6	+43.6	+42.0	4.5	25.7
Raspberry A	+123.6	+110.2	+103.5	2.6	62.8

employed, as well as their approximate proportion in the products examined.

Total Solids.—Ten grams of the jam, which has been evenly pulped in a mortar, or 5 grams of the jelly, are weighed into a tared platinum dish, taking care to spread the sample as thinly as possible over the bottom of the dish, and dried to nearly a constant weight at 100°. Results yielded by this method, while sufficiently close for ordinary work, are not exact, due to the slight dehydration of the sugars. If extreme accuracy is required, dry *in vacuo* at 75° C., or in a McGill oven, page 481.

Soluble and Insoluble Solids.—A weighed amount of the evenly pulped sample, say 25 grams, is vigorously shaken in a 500-cc. graduated flask with water, preferably with the aid of a mechanical shaker, and, after filling to the mark, is again shaken. The residue is allowed to settle, and the supernatant liquid is decanted through a filter, and an aliquot portion of the filtrate, say 50 cc., is measured into a tared dish and evaporated to dryness, dried at 100° to a constant weight, and weighed for soluble solids. Insoluble solids are calculated by difference.

Ash.—The residue from the total solids is burnt at dull redness to an ash, cooled in a desiccator, and weighed.

Nitrogen is determined by the Gunning method, page 61, in from 5 to 10 grams of the uniformly mixed sample.

DETERMINATION OF SUGARS.—In products of the highest grade, wherein only cane sugar is employed, a large portion of the cane sugar is inverted in the process of boiling the jam or jelly, so that when the analyst examines it, he finds, as a rule, only a small amount of sucrose, and considerable invert sugar. It is possible, however, to calculate the amount of cane sugar originally employed, if such information is desirable. It is further of interest to calculate, at least approximately, the percentage of commercial glucose, when present, especially in cases where the package contains a formula setting forth the amount of the various ingredients used. In such cases the analyst is naturally called upon to verify the formula, since a wide variation in percentage composition from the statement on the label would constitute an offense under some state laws.

Polarization.—Use half the normal weight of the preserve or jelly for the Schmidt and Haensch instrument, viz., 13.024 grams in 100 cc. If fresh fruit or fruit juice is to be examined, use the full normal weight, 26.048 grams. Clarify, before making up to the mark, with subacetate of lead and alumina cream (using 2 to 3 cc. of each clarifier), filter, and

obtain the direct reading; then invert in the usual manner, and obtain the invert readings at 20° C., and in the water-jacketed tube at 87° C., proceeding in detail as directed under honey, p. 515.

Calculation of Sugars.—*Sucrose* is determined by using Clerget's formula:

$$S = \frac{(a-b)100}{144-\frac{t}{2}}. \quad \ldots\ldots\ldots \quad (1)$$

This represents the sucrose actually present as such in the preserve or jelly, and not the amount originally used. If the latter is desired, it may be calculated from the formula,

$$S' = \frac{100b}{44-\frac{t}{2}}, \quad \ldots\ldots\ldots \quad (2)$$

where S' is the per cent of cane sugar originally used, and b is the invert reading at $t°$ of the normal solution.

If, after inversion, the correct reading at 20° is found to be 10 or more to the left of the zero, it can be safely inferred that no appreciable amount of commercial glucose is present, and it is unnecessary to make a third reading at 87°, unless to confirm the fact. In such a case, with cane sugar alone present, the reading at 87° will not, of course, vary much from 0.

Invert Sugar.—In the absence of commercial glucose, the invert sugar is calculated as follows:

$$\text{Invert sugar} = \frac{(\text{Sucrose} - \text{direct reading})105.3}{44-\frac{t}{}}, \quad \ldots \quad (3)$$



$$\text{Invert sugar} = \frac{\left(\begin{matrix}\text{Reading due to glucose and}\\ \text{inverted sucrose at } t^\circ\end{matrix}\right) - \left(\begin{matrix}\text{Invert reading}\\ \text{at } t^\circ\end{matrix}\right)}{\pm\left(44 - \frac{t}{2}\right)} 105.3 \quad (4)$$

These formulas, (3) and (4), serve at best to indicate the approximate amount of invert sugar present in the sample, resulting from the inversion of a portion of the original sucrose in the natural process of manufacture of the jam or jelly, and not the total invert sugar resulting from the inversion by the analyst of all the sucrose.

The factor 105.3 is used, since, in the natural process of inversion, 100 parts of sucrose become 105.3 parts of invert sugar.

Example.—The invert sugar in the sample of apple jelly first on the list in the table on page 718 is calculated as follows:

Invert reading at t° (20°) = 28.0.

Reading due to glucose at 20° = .221 × 175 = 38.68.

" " " inverted sucrose at 20° = .268 × −34 = −9.11.

$$\text{Invert sugar} = \frac{(38.68 - 9.11) - 28}{34} 105.3$$

$$= 4.86\%.$$

Reducing Sugar by Copper Reduction.* — Five grams of the preserve or jelly (or 25 grams of the fresh fruit or fruit juice) are transferred to a 100 cc. graduated flask, clarified by the addition of 2 or 3 cc. each of subacetate of lead and alumina cream, made up to the mark, shaken, and filtered. An aliquot part of the filtrate is then measured into another 100 cc. flask, and treated with enough of a saturated solution of sodium sulphate to precipitate the lead, after which it is made up to the mark and filtered. The amount of sugar solution measured off into the second flask is such that, when finally made up to 100 cc. as described, approximately ½ of 1% of reducing sugar is present, as roughly estimated by the total solids and polarizations. The reducing sugar is then determined in the filtrate as dextrose by Defren's method, page 480, or if Allihn's method is used (p. 403) the amount of reducing sugar present should approximate 1%.

Commercial Glucose.—While it is impossible to determine the exact percentage of this substance in preserves and jellies, by reason of the varying composition of its component parts, it is quite feasible to approximate very closely to the amount present. Indeed, this approximate

* U. S. Dept. of Agric., Bur. of Chem., Bul. 65, p. 78.

method of calculation, wherein glucose is treated as a chemical entity, has been found in practice to be much more close to the actual truth than results gained by methods wherein the copper reducing power enters as a factor, or methods for determining separately dextrin, maltose, and dextrose. Calculate the commercial glucose in jellies and jams exactly as in the case of honey, p. 514.

Dextrin.*—If alcohol be added to a somewhat thick solution of the fruit product, a white turbidity is at once apparent, followed by the formation of a thick gummy precipitate, if dextrin is present. In the absence of dextrin there is no turbidity, but a light flocculent precipitate.

To determine the dextrin, dissolve † 10 grams of the sample in a 100-cc. flask; add 20 mg. of potassium fluoride, and then about one-quarter of a cake of compressed yeast. Allow the fermentation to proceed below 25° C. for two or three hours to prevent excessive foaming, and then place in an incubator at a temperature of from 27° to 30° C. for five days. At the end of that time clarify with lead subacetate and alumina cream; make up to 100 cc. and polarize in a 200-mm. tube. A pure fruit jelly will show a rotation of not more than a few tenths of a degree either to the right or to the left. If a Schmidt and Haensch polariscope be used, and a 10% solution be polarized in a 200-mm. tube, the number of degrees read on the sugar scale of the instrument, multiplied by 0.8755, will give the percentage of dextrin, or the following formula may be used:

$$\text{Percentage of dextrin} = \frac{C \times 1000 \times V}{198 \times L \times W},$$

in which

C = degrees of circular rotation,

V = volume in cubic centimeters of solution polarized,

beaker with the glass stirring-rod to start the crystallization of the potassium bitartrate. Allow to stand fifteen hours at room temperature. Filter, and wash the precipitate into a Gooch crucible with a thin asbestos felt, using the vacuum pump. Wash with a mixture of 15 grams of potassium chloride, 20 cc. of alcohol, and 100 cc. of water. The beaker is rinsed three times with a few cubic centimeters of this solution. The precipitate is also washed with a few cubic centimeters, but so that not more than 20 cc. in all of the wash solution is used. The precipitate and asbestos filter are washed back into the beaker, and heated to boiling. While still hot, the solution is titrated with decinormal alkali, using phenolphthalein as indicator. To the amount of alkali used must be added 15 cc. for the potassium bitartrate remaining dissolved in the solution. 1 cc. of decinormal alkali is equivalent to 0.0150 grams of tartaric acid.

Determination of Citric Acid.*—Fifty cubic centimeters of the fruit solution is evaporated on the water-bath to a syrupy condition. To the residue add, very slowly at first, stirring constantly, 95% alcohol until no further precipitate is formed; 70 to 80 cc. are generally enough. Filter, and wash the residue with 95% alcohol. Evaporate the filtrate to eliminate the alcohol, take up the residue with a little water, and transfer to a graduated cylinder, making up to 10 cc. To 5 cc. of this solution, add half a cubic centimeter of glacial acetic acid, and to this add, drop by drop, a saturated solution of lead acetate. The presence of citric acid is shown by the appearance of a precipitate, which possesses the property of disappearing on being heated, and reappearing on cooling. In order to separate the citric acid from other acids, heat to boiling, filter, and wash with boiling water; then allow to cool, and the precipitate of lead citrate will re-form. This lead precipitate may be filtered off, washed with weak alcohol, dried, weighed, and the citric acid calculated. It is necessary that there shall be no tartaric acid present. If the tartaric acid has been estimated, any error on this account may be avoided by adding enough decinormal potash to neutralize the tartaric acid before the alcohol is added.

Detection of Coloring Matter.—Boil white woolen cloth or worsted in a solution of the jelly or jam, acidified with hydrochloric acid, or with acid sulphate of potassium, according to Arata's method and test for the color on the dyed fabric by methods given in detail in Chapter XVI.

* Moslinger, Zeit. Unter. Nahr. u. Genuss., 1899, 2, p. 93; U. S. Dept. of Agric., Bur. of Chem., Bul. 65, p. 80.

Detection of Preservatives and Concentrated Sweeteners.—Extract an acid aqueous solution of the fruit product with ether or chloroform in a separatory funnel, and test for benzoic and salicylic acids and for saccharin in the ether extract. If dulcin is suspected, extract with acetic ether.

Detection of Starch.*—Heat an aqueous solution of the preserve or jelly nearly to the boiling point, and decolorize by the addition of several cubic centimeters of dilute sulphuric acid and afterwards permanganate of potassium. This treatment does not affect the starch, which is tested for with iodine in the ordinary manner in the solution after cooling. In the clear filtrate from a boiled apple pulp solution, free from added starch, little or no darkening should occur on the addition of the iodine reagent. If, however, the reagent is added to the residue of the previously boiled pulp, the presence of starch inherent in the apple is usually recognized by the blue color produced thereon.

The presence of any considerable *added* starch paste in a fruit preparation is thus readily indicated by an intense blue color obtained by adding the iodine reagent to the filtrate (free from fruit pulp).

Detection of Gelatin.—*Robin's Method.*†—Add to a thick aqueous solution of the preserve or jelly sufficient strong alcohol to precipitate the gelatin. Decant the supernatant liquid after settling, set aside part of the precipitate, and dissolve the remainder in water. Divide the latter solution in two parts, to one of which add, drop by drop, a fresh solution of tannin, which precipitates gelatin if present. To the remainder add picric acid solution, which in presence of gelatin forms a yellow precipitate. The portion of the yellow precipitate set aside is transferred to a test tube, and heated over the flame with a little quicklime. If gelatin is present, ammonia will be given off, apparent by the odor, and by fumes



sediment is examined by the microscope for diatoms, which will be found in large numbers if agar agar has been used.

Detection of Apple Pulp.—A distinct clue to the presence of apple pulp in fruit preparations is often furnished by the characteristic apple odor given off when a small amount of the sample is heated to boiling with water in a test tube. Under such conditions, the apple odor is quite apparent, as distinguished from that of other fruits, especially if the apple is the chief fruit present, or predominates in the mixture.

Apple pulp in fruit preserves, free from added starch, may usually be recognized by a microscopical examination, using iodine reagent. The cell contents of the pulp will show the characteristic blue color, undoubtedly due to portions of unconverted starch still remaining in them.

Detection of Fruit Tissues under the Microscope.—It is a matter of some difficulty, by means of a microscopical examination, to identify with certainty the various fruits and vegetables that might be used in a jam or jelly as adulterants. The structural features of the common fruits, while possessing distinctive points of difference when examined separately and in the raw products, are so changed or broken down by the process of cooking, as to be with difficulty recognizable. The soft parenchyma which forms the main portion of the tissue of the fruit pulp is, as a rule, more or less disintegrated.

In the case of some of the smaller fruits, as the currant and raspberry, the cuticles resist the cooking process to such an extent as to show characteristic fragments, often recognizable in preserves and jellies under the microscope.

FRUIT JUICES.

Sweet cider, orange juice, lime juice, grape juice, raspberry shrub, and the juices of various other fruits and berries, may be so prepared and sterilized as to keep without fermentation when bottled, and are so put up in considerable variety, either with or without the addition of sugar.

Such preparations, if of the highest purity, should consist of the undiluted juices of these fruits, separated by pressure and carefully sterilized and bottled. They should contain no other fruit juice than that specified on their labels, and should be free from alcohol, added antiseptics, or coloring matter, unless the label specifies the presence of the added foreign materials. The addition of pure cane sugar to such prepara-

tions as grape juice is allowable, as well as charging with carbon dioxide to form so-called carbonated drinks.

The following analyses of pure fruit juices are taken from tables prepared by Winton, Ogden, and Mitchell, showing results on samples purchased in the Connecticut market, as well as on some samples made in the laboratory.*

	Solids.	Acids Other than CO_2 as Citric.	Cane Sugar.	Invert Sugar.	Polarization.			
					Direct.	After Inversion.	Temperature C.	Invert Reading at 87° C.
COMMERCIAL FRUIT JUICES.								
Blackberry	5.32	0.65	0.0	4.6	−1.3	−1.3	29.0	
Cherry	14.33	0.80	0.0	6.5	−1.9	−1.9	26.0	
Black currant	10.00	2.41	0.0	9.2	−2.7	−2.7	26.0	
Red currant	7.58	2.09	0.0	7.2	−2.1	−2.1	27.0	
Grape	15.29	0.91	0.0	21.7	−6.5	−6.5	25.0	−1.0
Lime fruit	7.78	6.50	0.0	0.0	0.0	0.0		
Orange	12.72	2.44	0.0	7.1	−2.1	−2.1	26.0	
Pineapple	8.07	0.81	1.5	5.1	0.0	−2.0	26.0	
Plum	10.81	1.00	0.0	0.3	−0.1	−0.1	26.0	
Quince	10.41	0.99	0.0	16.7	−5.0	−5.0	25.0	−2.2
Black raspberry	8.47	1.36	0.0	7.8	−2.3	−2.3	26.0	
Strawberry	5.69	0.99	0.0	5.1	−1.5	−1.5	26.0	
MADE IN LABORATORY.								
Peach	12.70	0.95	5.4	2.1	4.8	−2.2	28.0	0.0
Red raspberry	9.41	1.19	0.8	8.6	−1.6	−2.8	26.0	0.0
Blackberry	8.94	1.22	0.0	8.7	−2.4	−2.4	30.0	0.0
Huckleberry	11.40	0.51	0.6	16.7	−4.0	−4.8	30.0	−1.0
Pineapple	13.90	0.68	7.4	9.1	4.7	−4.8	28.0	−0.8

Antiseptics found most commonly in these preparations are boric, salicylic, benzoic, and sulphurous acids. Beta-naphthol should also be



Grape juice is prepared by sterilizing at a temperature of 80° the juice expressed from the crushed grapes, filtering by means of a press or otherwise, and sealing in carefully sterilized bottles. After bottling, a final sterilization is conducted at a temperature 5° below the first. Bottled grape juices are rarely carbonated.

Bottled Sweet Cider.—The composition of pure, freshly expressed apple juice is shown by the following table of analyses by Browne:*

	Specific Gravity.	Solids.	Invert Sugar.	Su-crose.	Total Sugar.	Total Sugar after Inver-sion.	Free Malic Acid.	Ash.	Unde-ter-mined (Pectin, etc.).	Left-handed Rotation Degrees Ventzke 400 mm. Tube.
Red astrachan.	1.0532	12.78	6.87	3.63	10.50	10.69	1.14	0.37	0.77	23.72
Early harvest.	1.0552	13.20	7.49	3.97	11.46	11.67	0.90	0.28	0.65	24.32
Yellow transparent.	1.0502	11.71	8.03	2.10	10.14	10.24	0.86	0.27	0.44	
Sweet bough.....	1.0498	11.87	7.61	3.08	10.69	10.85	0.10	.....	.....	30.40
Baldwin, green. ...	1.0488	11.36	6.96	1.63	8.59	8.68	1.24	0.31	1.22	36.16
" ripe.	1.0736	16.82	7.97	7.05	15.02	15.39	0.67	0.20	0.87	
Ben Davis.	1.0539	12.77	7.11	3.85	10.96	11.16	0.46	0.28	1.07	49.00

Bottled sweet cider, properly sterilized, should not differ materially from the fresh juice, and should contain no alcohol.

Salicylic acid is the antiseptic most commonly found in sweet bottled ciders examined by the writer.

Lime or Lemon Juice.—This, according to the U. S. Pharmacopœia, should consist of the freshly expressed juice of the ripe fruit of *Citrus limonum* (Risso), natural order of Rutaceæ. Our supply of both lemons and limes comes chiefly from the West Indies and the Mediterranean. Both varieties of the genus Citrus are used indiscriminately for furnishing commercial lime juice, though strictly speaking, only that of the lemon is recognized in the Pharmacopœia. The juice is sharply acid, and is largely composed of citric acid (about 7%), gum, sugar (3 to 4 per cent), and inorganic salts from 2 to 2½ per cent. It also usually contains a little lemon oil from the rind. According to the pharmacopœia, lemon juice (*Lemonis succus*) should conform to the following requirements:

"Specific gravity: not less than 1.030 at 15° C.

"It has an acid reaction upon litmus paper, due to the presence of about 7% of citric acid.

* Penn. Dept. Agric., Bul. 58, p. 29.

"On evaporating 100 grams of the juice to dryness, and igniting the residue, not more than 0.5 gram of ash should remain."

Of thirty samples of commercial lime juice examined in the Massachusetts State Board of Health laboratory, representing fifteen brands, all were deficient in citric acid, containing from 1.92 to 4.15 per cent, thus showing that these preparations are frequently watered. Fifteen were found to contain salicylic acid, seven had sulphurous acid, while two contained both these preservatives. Several were found colored with coal-tar dyes.

One sample examined by the author, purporting to be a "pure West Indian Lime Juice, triple refined," proved to be a mixture of hydrochloric and salicylic acids, colored with a coal-tar dye, and contained no lime juice whatever.

Acidity of lime juice is obtained by titrating 6.8 cc. of the sample against tenth-normal sodium hydroxide with phenolphthalein. The number of cubic centimeters of the standard alkali required, divided by 10, gives the per cent of citric acid present.

FRUIT SYRUPS.

Two classes of these preparations are on the market, one for use in soda-fountains, and one for "family trade," intended as a basis for sweetened drinks to be diluted with water and sugar. Some are made exclusively from pure fruit pulp and sugar, sterilized by heating and put up in tightly sealed bottles, while others of the cheaper variety are more apt to be entirely artificial both in color and in flavor, deriving the latter principally from the wide variety of artificial fruit essences now available. Commercial glucose is a frequent ingredient. The same classes of coal-tar dyes and antiseptics are found in these preparations as in the other



is comparatively insignificant. These two favorite essences are employed in nearly every household, and form a necessary adjunct to almost all forms of desserts, cakes, and confections, as well as to a wide variety of commercial preparations.

Vanilla Extract.

THE VANILLA BEAN is the source of pure vanilla extract, besides being used in chopped form directly as a flavoring agent. It is the fruit of the plant of the *Vanilla planifolia*, or flat-leaved vanilla. This climbing, perennial plant belongs to the orchid family, and is indigenous to Central and South America and the West Indies, but by far the highest prized beans are cultivated in Mexico. While different varieties differ in some details, the best cured beans of commerce, as a rule, are from 20 to 25 cm. in length and from 4 to 8 mm. thick, drawn out at their ends and curved at the base. They are rich dark brown in color, of a soapy or waxy nature to the touch, deeply rifted lengthwise, and covered with fine frost-like crystals of vanillin. When cut crosswise, the bean exudes a thick, odorless juice, containing calcium oxalate crystals.

The cross-section of the bean is ellipsoidal in shape. The thick brown walls inclose a triangular cavity, in which are the lobed placentas. Between these are papillæ, secreting a finely granular, yellow, balsam-like substance that contributes much to the flavor of the extract, and helps to give the cut bean its delicious odor.

When first gathered, the beans are yellowish green, fleshy, and without odor, developing their peculiar consistency, color, and smell by the process of fermentation or "sweating," which differs in various countries. According to the best methods the beans are sun-dried for nearly a month, being alternately pressed lightly between the folds of blankets, and exposed to the air. After the curing, they are packed in bundles.

Quicker methods of curing consist of the use of artificial heat and calcium chloride for drying, but the products so prepared are considered inferior in quality.

The Mexican vanilla beans are of the choicest grade, and command a high price, varying from ten to fifteen dollars per pound. The Bourbon beans, grown in the Isle of Réunion, are next in grade. These beans are shorter than the Mexican and much less expensive. They resemble the Tonka bean in odor. Beans from Seychelles and Mauritius are even shorter than the Bourbon beans, and are largely exported to England. The cheapest varieties are those from South America and Tahiti, which

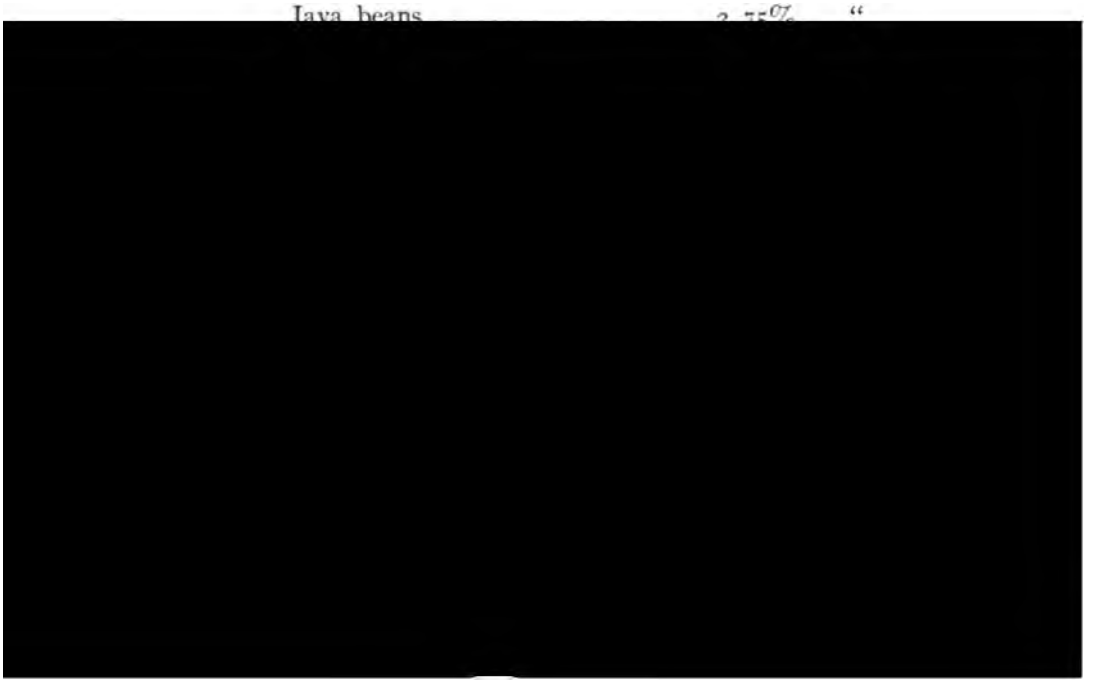
do not bring half the price of the Mexican beans, and the so-called "vanillons," or beans of the wild or uncultivated vanilla plants. These latter are used more in sachet powders and perfumes, possessing an odor not unlike heliotrope.

Composition of the Vanilla Bean.—The following are results of the analyses of two varieties of vanilla beans, according to König:

	A.	B.
Water	25.85	30.94
Nitrogen bodies	4.87	2.56
Fat and wax	6.74	4.68
Reducing sugar	7.07	9.12
Non-nitrogen substances	30.50	32.90
Cellulose	19.60	15.27
Ash	4.73	4.53

Vanillin.—Under "non-nitrogen substances" in the above table are included vanillin, the principle to which vanilla owes its peculiar odor. This body ($C_8H_8O_3$) is the methyl ether of protocatechuic aldehyde, and is found on the surface of the bean in fine crystalline needles. It has a sharp but pleasant flavor, is soluble with difficulty in cold water, but readily soluble in hot water, ether, alcohol, and chloroform. Its melting-point is 80° to 81° C. and it sublimes at 280°. It is present in vanilla beans to an amount varying from 1 to 2¾ per cent, and it is a curious fact that varieties of bean most highly prized possess the least vanillin. This is shown by Tiemann and Harmann as follows:

Mexican beans ..... 1.69% vanillin
Bourbon beans ..... 2.48% "
Java beans ..... 2.75% "



Exhausted Vanilla Beans are sometimes found on sale, which have been deprived of their vanillin by being soaked in alcohol, after which they are coated with some artificial substitute, presenting the same frosty appearance as the natural vanillin crystals. This may be accomplished by rolling the beans in benzoic acid. Benzoic acid crystals are readily distinguished from those of vanillin under the microscope.•

Composition of Vanilla Extract.—Vanilla extract is a dilute alcoholic tincture of the vanilla bean, sweetened by cane sugar. To be perfectly pure it should contain no other added substances, with the possible exception of glycerin, and many of the best brands are free from this. In practice it is variously prepared, but the following method of the U. S. Pharmacopœia is a typical one:

"Vanilla, cut into small pieces and bruised, 100 grams.

"Sugar, in coarse powder, 200 grams.

"Alcohol and water, each, a sufficient quantity to make 1,000 cc.

"Mix alcohol and water in the proportion of 650 cc. of alcohol to 350 cc. of water. Macerate the vanilla in 500 cc. of this mixture for twelve hours, then drain off the liquid and set it aside. Transfer the vanilla to a mortar, beat it with the sugar into a uniform powder, then pack it in a percolator, and pour upon it the reserved liquid. When this has disappeared from the surface, gradually pour on the menstruum, and continue the percolation, until 1,000 cc. of tincture are obtained."

The best extracts are produced by allowing the cut beans to macerate in the alcohol for several months.

Five vanilla extracts, made by Winton and Silverman from beans of different grades, strictly according to the pharmacopœial formula as above, were analyzed by them with the following results:

ANALYSES OF VANILLA EXTRACTS, U. S. P., MADE IN THE CONNECTICUT EXPERIMENT STATION LABORATORY.

Grade of Bean.	Specific Gravity.	Vanillin, Per Cent.	Alcohol by Weight, Per Cent.	Total Residue, Per Cent.	Cane Sugar, Per Cent.	Residue Other than Cane Sugar, Per Cent.
Mexican (whole)	1.0150	0.125	37.06	22.60	19.90	2.70
" (cut)	1.0140	0.065	39.02	23.10	19.20	3.90
South American (whole)	1.0100	0.215	38.58	22.00	19.00	3.00
Bourbon (whole)	1.0166	0.138	38.32	23.13	20.40	2.73
Tahiti (whole)	1.0104	0.108	38.84	21.75	20.00	1.75

Vanillin Content.—The writer has found in his examination of a large number of brands of vanilla extract that gave every indication of

purity that the content of vanillin varied from 0.05 to 0.200 per cent. It is rare that a pure extract will show more vanillin than the latter figure, though one of Winton's extracts runs as high as 0.215. The writer has found extracts showing 0.250 of vanillin, but believes them to have been reinforced with the artificial substance. It is true that the vanillin content does not determine altogether the value of the extract, which depends quite as much for its virtue as a flavor upon the various resinous and other extractive matters which it contains, as upon the actual amount of vanillin present.

Use of Alkali.—Some manufacturers employ dilute alkali, generally potassium bicarbonate, to aid in dissolving out the resinous matter from the bean, and to enable them to use a more dilute alcohol. The resulting product made by this process is distinctly inferior, both in taste and odor.

Alcohol in pure extracts varies between the limits of 20 and 40 per cent.

THE TONKA BEAN forms the basis of many of the cheaper so-called vanilla extracts on the market. It is the seed of the large tree, native to Guiana, known as *Dipterix* (or *Coumaronna*) *odorata*. The pods are almond-shaped, and contain a single seed, from 3 to 4 cm. long, shaped like a kidney bean, of a dark-brown color, having a thin, shiny, rough, brittle skin, and containing a two-lobed oily kernel.

Coumarin ($C_9H_6O_2$), the active principle of the Tonka bean, is the anhydride of coumaric acid. It occurs in the crystalline state between the lobes of the seed kernel. Coumarin occurs also in many other plants. It may be extracted from the beans by treatment with alcohol. It crystallizes in slender, colorless needles, melting at 67° C. It has a fragrant odor and burning taste. It is very slightly soluble in cold water, but readily soluble in hot water, ether, chloroform, and alcohol. One pound of cut beans yields by alcoholic extraction about 108 grains of coumarin.



The Adulteration of Vanilla Extract consists chiefly in the use of coumarin or extract of the Tonka bean, and in the substitution of artificial vanillin, either alone or with coumarin, for the true extractives of the vanilla bean. The cheaper, or so-called "compound" vanilla extracts, more often consist of a mixture of either tincture of Tonka or coumarin with vanillin in weak alcohol, colored with caramel. Or the exhausted marc from high-grade vanilla extract is macerated with hot water and extracted, the extract being reinforced with artificial vanillin or coumarin, or both. A pure vanilla extract possesses certain peculiarities with regard to its resinous content that at once distinguishes it from the artificial, or indicates whether or not it has been tampered with. While it is possible to introduce artificial resinous matter in the adulterated brands, with a view to deceiving the analyst, it is almost impossible to do this without detection, since different reactions are readily apparent in this case from those of the pure extracts.

Prune juice is said to be used to give body and flavor to vanilla extract. The writer has found spirit of myrcia or bay rum in a sample of alleged vanilla extract, containing also vanillin and coumarin. The adulterant in this sample was present to such an extent as to be unmistakable by reason of the odor.

Factitious Vanilla Extracts are ordinarily indicated (1) by the presence of coumarin, (2) by the peculiar reactions of the resinous matter, or by the entire absence of these resins, and (3) by the abnormally low content, or absence of vanillin.

The following figures show the content of vanillin and coumarin in a few typical cheap "vanilla" extracts, selected from a large number examined by the author. All of these were entirely artificial, and ranged from 5 to 20 per cent by weight of alcohol.

	Vanillin, Per Cent.	Coumarin, Per Cent.
A.	0.040	0.074
B.	None	0.172
C.	None	0.330
D.	0.250	None
E.	0.025	0.144

As a rule these cheap artificial preparations possess considerable body and flavor, but the latter is of a much grosser nature than the genuine vanilla extract, with the delicate and refined flavor of which they are not to be mistaken by any one at all familiar with both varieties.

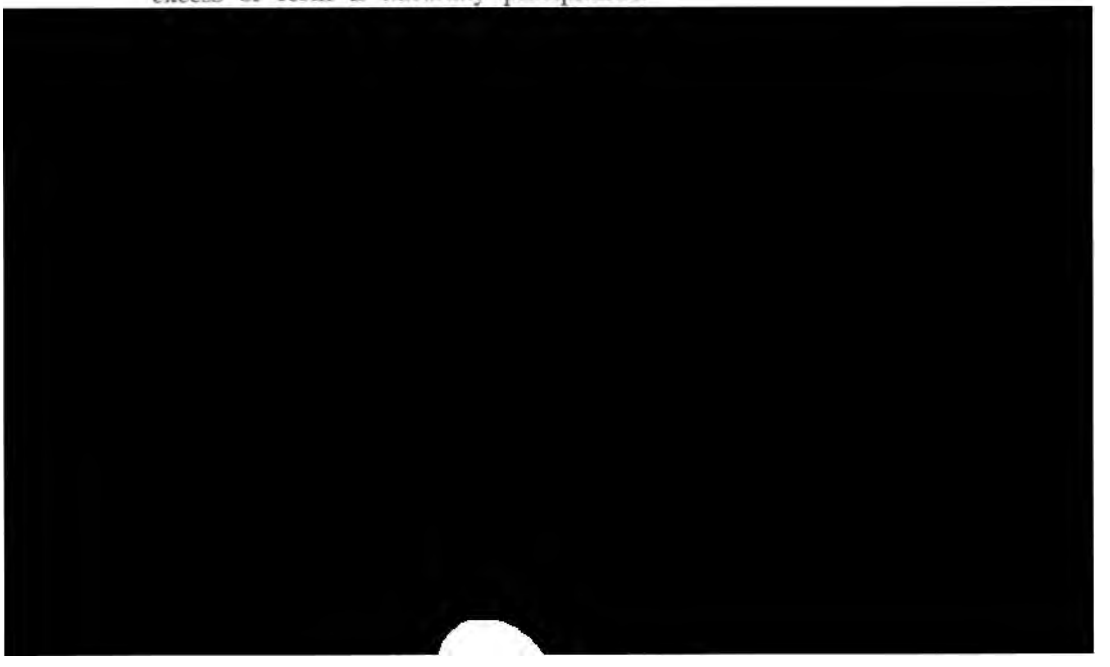
The author is inclined to place 0.05% of vanillin as a minimum for genuine vanilla extract properly prepared, and makes a practice of classing as not of good standard quality those samples that fall below.

Detection of Artificial Extracts.—The presence of coumarin or Tonka tincture to any appreciable extent in vanilla extract is usually recognizable by the odor, to one skilled in examining these flavors. The odor of coumarin is more pungent and penetrating than that of vanillin, and in mixtures is apt to predominate over the milder and more delicate odor of vanillin.

Add normal acetate of lead solution to a suspected extract. The *absence* of a precipitate is conclusive evidence that it is artificial. If a precipitate is formed, much information may be gained by its character. A pure vanilla extract should yield with lead acetate a heavy precipitate, due to the various extractives. The precipitate should settle in a few minutes, leaving a clear, supernatant, partially decolorized liquid. If only a mere cloudiness is formed, this may be due to the caramel present, and in any event is suspicious.

Examination of the Resins.—Resin is present in vanilla beans to the extent of from 4 to 11 per cent, and the manufacturer of high-grade essences endeavors to extract as much as possible of this in his product. This he can do by the use of 50% alcohol, in which all the resin is readily soluble, or by employing less alcohol and relying on the use of alkali to dissolve it. A pure extract free from alkali should produce a precipitate, when a portion of the original sample is diluted with twice its volume of water and shaken in a test-tube.

When, moreover, the alcohol is removed from such an extract, the excess of resin is naturally precipitated.



ing, drying, and weighing, but in this case should stand for a long time before filtering.

The resin is collected on a filter, washed, and subjected to various tests. A piece of the filter with the attached resin is placed in a beaker, containing dilute potassium hydroxide. Pure vanilla resin dissolves to a deep-red color, and is reprecipitated on acidifying with hydrochloric acid. Dissolve another portion of the precipitate in alcohol, and divide the alcoholic solution into two portions, to one of which add a few drops of ferric chloride, and to the other hydrochloric acid. Pure vanilla resin shows no marked coloration in either case, but foreign resins nearly all give color reactions under these conditions.

Tannin.—Test a portion of the filtrate from the resin for tannin by the addition of a few drops of a solution of gelatin. A small quantity of tannin only should be indicated, if the extract is pure, a large excess tending to show added tannin.

Determination of Vanillin.—Vanillin may be determined (1) colorimetrically, or (2) by extraction and weighing. The former is by far the quicker and more economical method, since it may be carried out directly in a very small portion of the original alcoholic extract. When, as in some instances, the analyst has only one small bottle of vanilla extract for analysis, it becomes a matter of importance to use as little as possible for each determination. The determination of vanillin by both methods should give concordant results.

(1) *Colorimetric Method.**—This is carried out in the author's laboratory as follows: 2 cc. of the vanilla extract are measured into a test-tube, and sufficient lead hydrate is added to completely decolorize it. The mixture is washed upon a filter, and filtrate and washings are collected in a Nessler tube. Bromine water is then added, after which enough of a freshly prepared 10% ferrous sulphate solution is added to get the maximum bluish-green color that will be produced, if vanillin is present. A standard vanillin solution is freshly prepared by dissolving 50 mgm. of pure vanillin in 25 cc. of alcohol, and making up to 100 cc. with water. A series of color standards is then made, taking, for instance, $\frac{1}{2}$, 1, $1\frac{1}{2}$, 2, $2\frac{1}{2}$, 3, etc., cc. of the vanillin solution in 50 cc. Nessler tubes, each being treated with two or three drops of bromine water, and with the ferrous sulphate solution, and made up to the 50-cc. mark.

The lead hydrate is prepared by dissolving 200 grams of lead acetate in 850 cc. of water. The solution is filtered, a solution of potassium

* Massachusetts State Board of Health, An. Rep., 1899, p. 629.

hydroxide is added in excess, and the precipitated hydrate is washed thoroughly several times by decantation, or until neutral. Keep an excess of water in the reagent bottle, and shake up to form a heavy, white emulsion before adding to decolorize. Unless the lead hydrate is washed free from alkali, the latter will precipitate the iron salt when added.

If, for example, 2 cc. of a sample extract, treated as above, are found to give a color corresponding in depth to that produced by 5.5 cc. of the standard solution, the percentage of vanillin would be thus calculated:

100	cc.	standard	solution	contain	0.050	gram	vanillin.
1	cc.	"	"	"	0.0005	"	"
5.5	cc.	"	"	"	0.00275	"	"

Since 2 cc. of the sample are equivalent to 5.5 cc. of the standard solution, it follows that

2 cc. of sample contain 0.0275 gram vanillin.
∴ 100 cc. " " " 0.1375 " "

In order to avoid calculation of each determination when a large number of extracts have to be examined, the following table will be found useful for expressing results, where the above method of procedure has been exactly carried out:

Number of Cubic Centimeters of Standard Vanillin Solution * Corresponding to 2 cc. of Sample.	Equivalent Per Cent of Vanillin in Sample.
0.25	0.00625
0.5	0.0125
0.75	0.01875
1.	0.025
1.5	0.0375



(2) *Extraction Method.**—Drive off the alcohol from 25 grams of the sample in an evaporating-dish on the water-bath at a temperature not exceeding 80° C., occasionally adding water to keep up about the original volume. After de-alcoholizing, add lead acetate drop by drop till all precipitable matter is thrown down, stir to aid the gathering of the precipitate, filter, and wash with three portions of hot water. Cool the filtrate, and extract with three or four portions of 20 cc. each of ether in a separatory funnel, stopping the extraction when no residue is left on evaporating a few drops of solvent in a watch-glass. The combined ether extract, which contains the vanillin and coumarin, is transferred to a separatory funnel, and shaken out several times with 10 cc. of 2% ammonia. This extracts the vanillin, leaving the ether solution of coumarin, if present.

Acidulate slightly the combined ammoniacal extracts with weak hydrochloric acid, and, after cooling, extract in a separatory funnel with ether till exhausted of its vanillin, evaporate off the excess of ether spontaneously in a tared platinum dish, dry over sulphuric acid in a desiccator, and weigh. Identify the residue as vanillin by the melting-point (81°–82° C.), by odor, by appearance of the crystals both megascopically and microscopically, and by testing in aqueous solution with ferric chloride, which should yield a dark-green coloration. If, however, the form of crystallization is not conclusive, or if the crystals are colored or accompanied by amorphous matter, without disturbing the residue, extract it in the dish with various portions of boiling petroleum ether till all the vanillin is removed, decanting each portion into a dry beaker.

The dish is again dried, this time at 100°, and weighed. If any residue is found, it is deducted from the first figure. The second treatment is rarely found necessary.

Determination of Coumarin.—Transfer the original ether solution (from which the vanillin was removed by ammonia) to a tared platinum dish, evaporate off the ether at room temperature over sulphuric acid in a desiccator, and weigh. Here, as in the case of vanillin, the character and appearance of the crystals will indicate whether or not foreign matter is present with the coumarin. If pure, the crystals should be perfectly white and well formed, and no dark-colored matter should be present. The odor of the pure coumarin is unmistakable, and the melting-point (67° C.) as well as the microscopical appearance may serve to identify

* Winton's Modification of Hess and Prescott's Method, Jour. Am. Chem. Soc., 21, 1899, p. 257; *ibid.*, 24, 1902, p. 1129.

it. If there is doubt as to the purity of the first residue, without disturbing it by tests, extract with boiling petroleum ether, and correct in the same manner as directed for vanillin.

In case the colorimetric method for vanillin was used, and coumarin only is to be separated for gravimetric determination, the author has found that good results are usually obtained by simply treating the dealcoholized original sample with ammonia, extracting it with 3 or 4 portions of chloroform in a separatory funnel, and evaporating the combined chloroform extract in a tared dish at a temperature not exceeding 60° in the oven.

If the residue is small, or if in any case there is some doubt as to whether or not any coumarin is present therein, apply the author's test for coumarin as follows: Add a few drops of water, warm gently, and add to the solution a little iodine in potassium iodide, reagent No. 143. In presence of coumarin a brown precipitate will form, which on stirring with the rod will soon gather in dark-green flecks. The reaction is especially marked if done on a white plate or tile. Many of the precautions employed in carrying out the above processes for vanillin and coumarin determination may be dispensed with, if these substances are simply to be tested for qualitatively.

Vanillin and Coumarin Crystals Under the Microscope.—These substances are best examined when crystallized from ether solution, and several crystallizations may be found necessary, before the best results are obtained. For examination, pour a few drops of the ether solution of the purified vanillin or coumarin directly on a slide, and allow to evaporate spontaneously. Under best conditions vanillin crystallizes from ether in long, slender needles, often radiating from central points, or forming star-shaped bundles.



To determine glycerin, proceed as with wines (p. 570).

Determination of Alcohol.—Measure out 25 cc. of the sample, dilute to 50 cc. with water, and distill off about 20 cc. in a 25-cc. graduated receiver. Make up to the mark with water, determine the specific gravity at 15.6°, and find from the alcohol table the per cent corresponding.

Cane Sugar and Glucose are determined as in the case of preserves and jellies.

Caramel is best detected by Crampton and Simons' method with fullers' earth (p. 603).

Lemon Extract.

Lemon extract, the *Spiritus limonis* or essence of lemon of the Pharmacopœia, is directed to be made in the following manner:

"Oil of lemon..... 50 cc.
"Lemon peel freshly grated..... 50 grams
"Deodorized alcohol, a sufficient quantity to make 1000 cc.

"Dissolve the oil of lemon in 900 cc. of deodorized alcohol, add the lemon peel, and macerate for twenty-four hours. Then filter through paper, and add, through the filter, enough deodorized alcohol to make the spirit measure 1000 cc."

The standard fixed by the Pharmacopœia (5% of lemon oil by volume) is a fair one. In fact there are commercial extracts on the market containing as high as 9%. An extract of lemon to contain 5% of lemon oil must contain at least 80% by volume of alcohol, lemon oil being insoluble in dilute alcohol. Deodorized, or purified alcohol, commonly known as cologne spirits or perfumers' alcohol, is used in the highest-grade preparations, since the odor of ordinary commercial alcohol produces a slightly deleterious effect.

Though there are many reputable brands of lemon extract on sale containing 5% or more of lemon oil, the market is flooded with the cheaper "ten-cent" variety, many purporting to be pure, others marked simply "compound," and others again with formulas having the name and per cent of the ingredients.

The classification of these extracts, whether as pure or adulterated, depends largely on the local laws in any particular state.

Adulteration of Lemon Extracts.—For making a cheap extract the cost of the lemon oil is not so important an item as that of the alcohol, and as little as possible of the latter is employed, though pure oil is doubt-

less used. The average cheap extract is made by rubbing the oil in carbonate of magnesia in a mortar, stirring in slowly a little strong alcohol, and allowing the mixture to soak for some time. A varying amount of water is added a little at a time, and the whole is shaken and again allowed to stand, sometimes for a week, before filtering. Finally the extract is filtered and the coloring matter added, consisting sometimes of turmeric tincture and sometimes of coal-tar dyes. In these cheap extracts the per cent of alcohol often runs below 40, and as little as 4.5% by volume of alcohol has been found by the author in a commercial extract. With less than 45% of alcohol by volume, no appreciable amount of oil is dissolved, only the merest traces, though such preparations are frequently bottled as "pure extract of lemon." Time and again manufacturers have protested to the author that the purest oil was used by them, when notified that their brand contained no oil, or when prosecuted in court, and were with difficulty convinced that the trouble with their goods was that, on account of weak alcohol employed, the lemon oil used failed to get into the final product. It is true that a certain taste or odor of the lemon is present, even in the cheap varieties wherein no oil is found, due to the fact that even dilute alcohol, when slowly percolating through the magnesia in which the oil is finely distributed, does abstract therefrom a suggestion of the flavor, which is, however, but a mere shadow of the substance and body possessed by a strong alcoholic solution of oil of lemon.

In many instances, where formulas appear stating the name and per cent of ingredients, these formulas are entirely deceptive and misleading, in that the statements are not borne out on analysis.

Few manufacturers, for example, are prepared to state boldly that their product contains no oil of lemon, a condition which in this class



chusetts were collected and analyzed. 167 samples were examined, representing about 100 brands, and 139 samples were classed as adulterated, based on 5% lemon oil as a standard, and depending on whether or not the contents conformed to the labels on the bottles.

The following are typical analyses, selected from the tabulated results of the above examination:*

LEMON EXTRACTS OF STANDARD QUALITY.

Polarization in 200-mm. Tube.	Lemon Oil, Per Cent by Volume.	Specific Gravity at 15.6° C.	Alcohol, Per Cent by Volume.	Foreign Ingredients.
30.8	9.1	0.8280	84.39	Turmeric
26.0	7.6	0.8402	80.49	
23.5	6.9	0.8352	81.74	Dinitrocresol
21.8	6.4	0.8396	82.88	
20.0	5.9	0.8335	84.24	
18.0	5.3	0.8268	86.82	
17.0	5.0	0.8496	80.06	

INFERIOR OR ADULTERATED LEMON EXTRACTS.

Polarization in 200-mm. Tube.	Lemon Oil, Per Cent by Volume.	Specific Gravity at 15.6° C.	Alcohol, Per Cent by Volume.	Foreign Ingredients.
14.0	4.1	0.8592	77.62	Dinitrocresol
12.2	3.6	0.8644	76.08	"
11.0	3.1	0.8620	77.50	A coal-tar dye
9.9	2.9	0.8615	77.90	"
8.0	2.3	0.8531	81.61	Dinitrocresol
6.8	2.0	0.8416	87.55	Tropæolin
5.0	1.5	0.8832	71.10	"
3.5	1.0	0.8939	67.68	
2.8	0.8	0.8995	65.23	Dinitrocresol
2.2	0.6	0.8941	67.69	"
1.4	0.4	0.9136	59.40	A nitro dye
0.3	0.1	0.9408	46.40	Dinitrocresol
0.0	0.0	0.9937	4.49	Tropæolin
−8.0	0.0	.....	.....	Invert sugar
27.0	0.0	.....	27.49	Cane sugar
0.0	0.0	.....	47.35	Oil other than lemon

Forty-two samples contained no lemon oil, ranging in content of alcohol from 4 to 45 per cent.

Methods of Analysis. A. S. Mitchell was the earliest worker who systematically examined lemon extract, and to him are due the methods for determining oil of lemon, as well as various other tests now adopted provisionally by the A. O. A. C.†

* An. Rep. Mass. State Board of Health, 1901, p. 459; Food and Drug Reprint, p. 41.

† Jour. Am. Chem. Soc., 21, 1899, p. 1132; U. S. Dept. of Agric., Bur. of Chem., Bul. 65, p. 73.

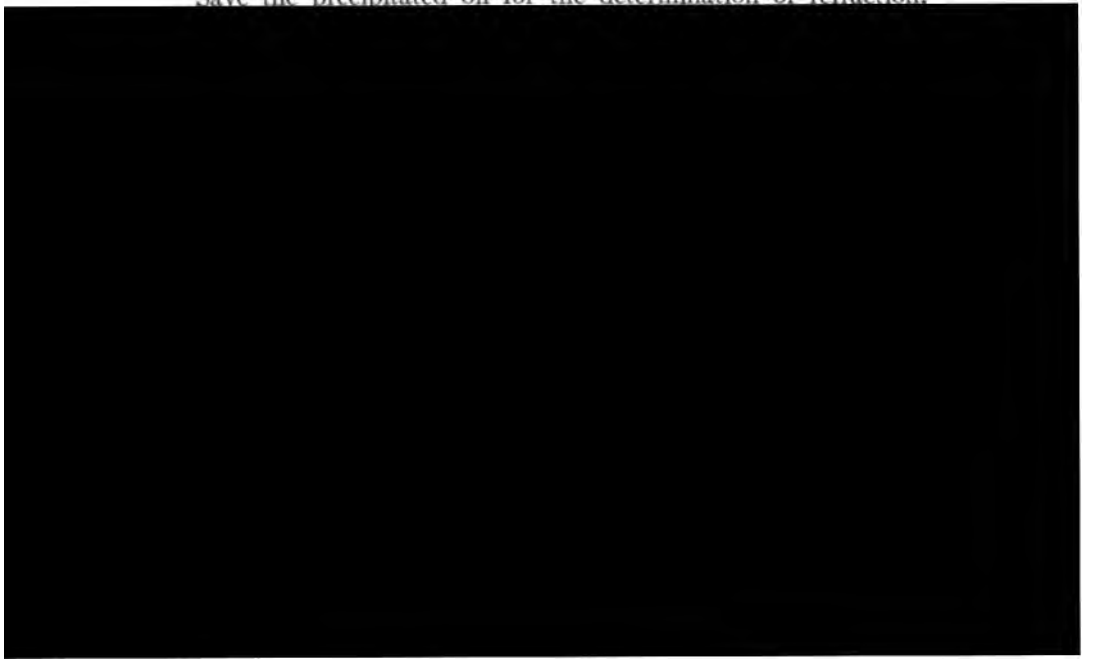
Detection of Oil of Lemon.—If on adding a large excess of water to a little of the extract in a test-tube no cloudiness occurs, the oil may fairly be inferred to be absent. The degree of cloudiness produced is proportional to the amount of lemon oil present.

Determination of Lemon Oil.—*Mitchell's Methods.*—(1) *By Polarization.*—Polarize the undiluted extract in a 200-mm. tube at 20° C. Divide the reading on the Ventzke cane sugar scale by 3.4, and if cane sugar or other optically active substances are absent, the quotient expresses the per cent of lemon oil by volume. With instruments reading in circular degrees, divide the rotation in minutes at 20° C. by 62.5. If the Laurent instrument with sugar-scale is used, divide the sugar-scale reading by 4.8.

Cane sugar, though rarely found in lemon extract, is occasionally used in small amount. It is said to aid in the solution of the oil. If it is present, wash the solid residue from 10 cc. of the sample (dried on a water-bath) with three portions of 5 cc. each of ether, to remove waxy and fatty matters, dry and weigh the residue of cane sugar, deducting 0.38 from the reading for each 0.1% of sugar so found.

(2) *By Precipitation.*—Transfer by a pipette 20 cc. of the extract to a Babcock milk-flask, add 1 cc. of dilute hydrochloric acid (1 : 1); add 25 to 28 cc. of water previously warmed to 60° C.; mix, and stand in water at 60° for five minutes; whirl in a centrifuge for five minutes; fill with warm water to bring the oil into the graduated neck of the flask, and repeat the whirling for two minutes; stand in water at 60° for a few minutes, and read the per cent of oil by volume. Where the oil of lemon is present in amounts over 2%, add to the percentage of oil found 0.4% to correct for the oil retained in solution. Where less than 2% and more than 1% is present, add 0.3% for correction.

Save the precipitated oil for the determination of refraction.



gravity of the sample, ascertain from the alcohol tables on pages 531, 544, the per cent of alcohol by volume corresponding. This gross figure includes the lemon oil, the per cent of which should be deducted for the correct per cent of alcohol.

In the absence of oil of lemon, a measured portion of the original sample may be distilled, and the percentage of alcohol determined from the distillate in the usual manner, but when lemon oil is present, this should first be removed by diluting 50 cc. of the extract to 200 with water, and shaking the mixture with 5 grams of magnesium carbonate in a flask, filtering through a dry filter, and determining the alcohol by distillation in a portion of the filtrate. The result is multiplied by 4 to correct for the dilution.

Methyl Alcohol has been used by unscrupulous manufacturers in lemon extracts. Methyl alcohol is best detected by the method of Mulliken and Scudder,* depending on conversion of the methyl alcohol to formaldehyde.

Dilute a portion of the distillate obtained in the determination of alcohol (or, for preliminary examination, dilute and filter the original extract) until the liquid contains approximately 12% of alcohol by weight.

Oxidize 10 cc. of the liquid in a test-tube as follows: Wind copper wire 1 mm. thick upon a rod or pencil 7 to 8 mm. thick, in such a manner as to inclose the fixed end of the wire and to form a close coil 3 to 3.5 cm. long. Twist the two ends of the wire into a stem 20 cm. long, and bend the stem at right angles about 6 cm. from the free end, or so that the coil may be plunged to the bottom of a test-tube, preferably about 16 mm. wide and 16 cm. long. Heat the coil in the upper or oxidizing flame of a Bunsen burner to a red heat throughout. Plunge the heated coil to the bottom of the test-tube containing the diluted alcohol. Withdraw the coil after a second's time and dip it in water. Repeat the operation from three to five times, or until the film of copper oxide ceases to be reduced. Cool the liquid in the test-tube meanwhile by immersion in water.

Test for Formaldehyde.—Divide the oxidized liquid in the test-tube into two parts, testing one for formaldehyde with pure milk by the hydrochloric acid and ferric chloride test. Test the other portion by Mulliken and Scudder's resorcin test for formaldehyde, page 666, avoiding an excess of the reagent.†

* Am. Chem. Jour., 21, 1899, p. 266.

† *Ibid.*, 24, 1900, p. 451.

Tests for Colors.—Evaporate a portion of the sample to dryness, dissolve the residue in water, and extract coal-tar colors if present by Arata's method, page 641, or with hydrochloric acid.

Much information may often be gained by treatment of the original extract with strong hydrochloric acid. If the color employed be turmeric or naphthol yellow, no change in color will be evident on addition of the acid. If tropæolin or methyl orange is present, the solution will turn pink, while partial decoloration of the solution indicates Martius yellow, and complete decoloration shows presence of dinitrocresols.

Test for turmeric by boric acid, page 637.

Determination of Total Solids and Ash.—*Total Solids* are estimated by evaporating on the water-bath 10 grams of the sample in a tared dish, and drying at 100° to constant weight. If glycerin be present, it is difficult if not impossible to get a constant weight. Cane sugar and glycerin, if present, will be apparent in the residue. If capsicin has been used, it will be noticed by the taste.

Burn to an ash the residue from the solids in a muffle at a low red heat, cool in a desiccator, and weigh.

Glycerin is determined as in wine, page 570.

Detection of Tartaric or Citric Acid.—To a portion of the extract in a test-tube add an equal volume of water to precipitate the oil. Filter and add one or two drops of the filtrate to a test-tube half full of cold, clear lime water. If tartaric acid is present, a precipitate will come down, which is soluble in an excess of ammonium chloride or acetic acid.

Filter off the precipitate, or, if no precipitate is visible, heat the contents of the tube. Citric acid will precipitate in a large excess of hot lime water.

Examination of Lemon Oil.—The oil separated from the extract in

READINGS ON ZEISS BUTYRO-REFRACTOMETER OF LEMON OIL.

Temperature, Centigrade.	Scale Reading.	Temperature, Centigrade.	Scale Reading.	Temperature, Centigrade.	Scale Reading.	Temperature, Centigrade.	Scale Reading.
40.0	59.4	35.0	62.8	30.0	66.3	25.0	69.7
39.5	59.7	34.5	63.1	29.5	66.6	24.5	70.0
39.0	60.1	34.0	63.5	29.0	67.0	24.0	70.4
38.5	60.4	33.5	63.8	28.5	67.3	23.5	70.7
38.0	60.8	33.0	64.2	28.0	67.7	23.0	71.1
37.5	61.0	32.5	64.5	27.5	68.0	22.5	71.4
37.0	61.5	32.0	64.9	27.0	68.4	22.0	71.8
36.5	61.8	31.5	65.1	26.5	68.7	21.5	72.1
36.0	62.1	31.0	65.6	26.0	69.0	21.0	72.5
35.5	62.4	30.5	65.9	25.5	69.3	20.5	72.8
35.0	62.8	30.0	66.3	25.0	69.7	20.0	73.2

For examination of high polarizing essential oils like oil of lemon, the author employs a 50-mm. tube, in order to get readings on the undiluted oil well within the limits of the cane sugar scale on the polariscope. If such a tube is not available, dilute the oil with an equal volume of alcohol, and use the 100-mm. tube. The following table expresses constants of pure lemon oils and of various commonly employed adulterants, as determined in the laboratory of the Massachusetts State Board of Health:

CONSTANTS OF SOME ESSENTIAL OILS.

Oil.	Butyro-refractometer (Sodium Light) at—		Rotation in 100-Millimeter Tube, Ventzke Scale.	Specific Gravity at 15.0° C.
	Temp.	Reading.		
Oil of lemon (lowest)	25.	69.5	173.0	0.8580
" " " (highest)	25.	71.2	184.5	0.8610
" " " grass (A. Giese)	22.5	96.9	−10.8	0.0309
" " citronella (A. Giese)	22.5	87.1	−10.2	0.0437
Terpeneless oil of lemon (Hansel's)	23.	87.9	−22.0	0.0463
" " " " grass (Hansel's)	23.	91.0	−5.6	0.0232
Citral (A. Giese)	22.5	95.0	−3.6	0.0296

Oil of Lemon is a light-yellow liquid, having the pleasant odor of fresh lemons, and an aromatic, mild, somewhat bitter after taste. It is obtained from the grated rind of the lemon either by treatment with hot water, skimming off the oil which rises to the surface, or by pressure, or by distillation with water. It is rapidly changed by action of air and light, becoming "terpeney," and under these conditions its solubility in alcohol seems to increase. Its composition is somewhat uncertain, but according to Wallach * nearly 90% consists of hydrocarbons, mostly

* Liebig's Annalen, 227, p. 290.

terpenes, the most important of which is the terpene limonene * of the dextro-gyrate variety, also known as citrene.

Another important constituent of lemon oil is the aldehyde citral, present to the extent of from 7 to 10 per cent. To this the odor of the oil is largely due. A second aldehyde, citronellal, is also present in lemon oil.

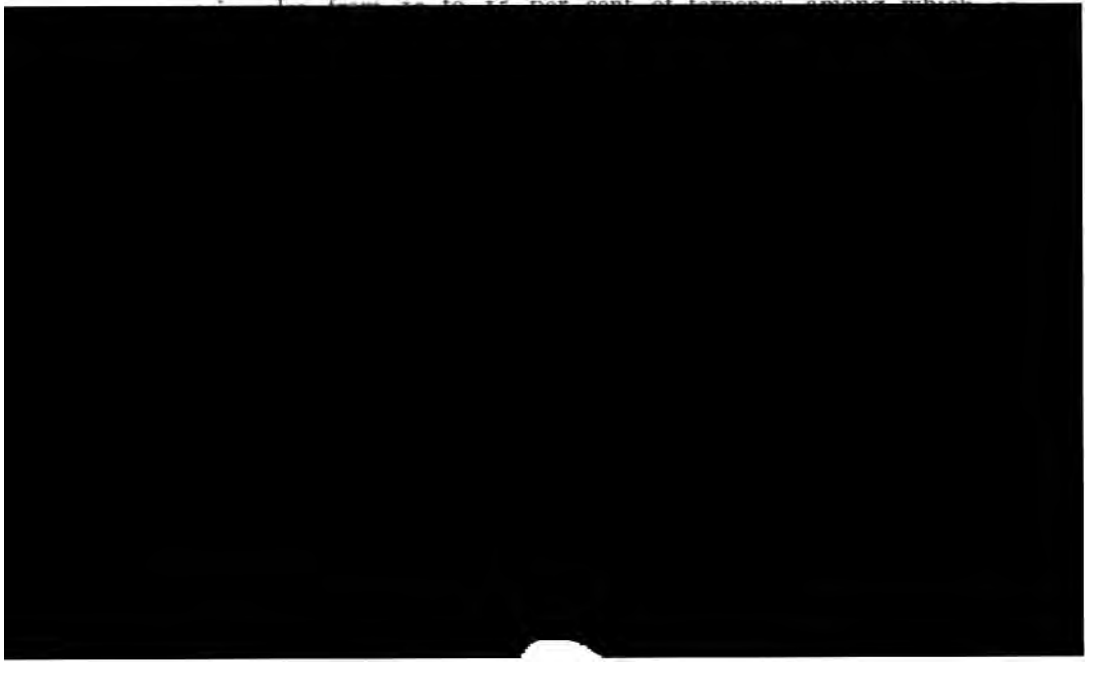
A frequent adulterant of lemon oil is turpentine oil, which lowers the rotation considerably, and is thus most easily rendered apparent.

Citral ($C_{10}H_{16}O$) is an aldehyde present in lemon oil and in oil of lemon-grass, and, while it may be separated from these oils, is prepared artificially by oxidizing geraniol with chromic acid.† It is a mobile oil, and when perfectly pure is optically inactive. The commercial citral is, however, slightly lævo-rotary, due no doubt to impurities.

Oil of Lemon-grass is distilled from lemon-grass, *Andropogon citratus* (D. C.), cultivated in India. It is reddish yellow in color, and has an intense lemon-like odor and taste. Very little is known of its composition, but it seems to contain several aldehydes, one of which is citronellal, and another citral. The latter, however, is its chief constituent, being present to the extent of 70 to 75 per cent.

Citronellal ($C_{10}H_{18}O$) is an aldehyde found in various oils, especially in citronella oil, from which it is readily separated. It is made artificially by the oxidation of the primary alcohol citronellol ($C_{10}H_{20}O$). It is quite strongly dextro-rotary.

Oil of Citronella is distilled from the grass *Andropogon nardus* (L.), growing chiefly in Ceylon, India, and tropical East Africa. It is a yellowish-brown liquid with a pleasant and lasting odor. Citronellal is present in this oil to the extent of from 10 to 20 per cent, and the oil



Limonene forms an evanescent, faint flesh color, and leaves a white compound.

MISCELLANEOUS FLAVORING EXTRACTS.

A much larger variety of flavors are used in confectionery and soda-water syrups than are required by the housewife in ordinary cooking. These miscellaneous flavors may be divided into two classes, first those which, like lemon essence, are alcoholic extracts of oils, including such essences as orange, clove, nutmeg, ginger, mace, cassia, peppermint, spearmint, wintergreen, and almond; and second, those which are made up of artificial fruit essences or compound ethers, which are ingeniously mixed to imitate the pure fruits. Under this class are included such flavors as strawberry, raspberry, pineapple, cherry, banana, pear, rose, peach, plum, apricot, and quince.

In the case of extracts of oils, it is generally practicable to separate the oil for examination by the centrifuge, as in the precipitation method of Mitchell and to estimate the same directly as described on page 742, when the specific gravity of the oil in question is less than water. In the case of such oils as clove, wintergreen, and cassia, where the specific gravity is greater than water, it is necessary to add salt to the extract before centrifuging, to cause separation of the oil.

The following table shows the specific gravity and polarimetric reading of these "essence" oils:

	Specific Gravity.	Polarization 100 mm, Ventzke Scale.	n_D at 15° C.
Orange	0.848 to 0.852	276.8 to 282.6	1.4774
Clove	1.045 " 1.070	−3.8	1.5245
Nutmeg	0.865 " 0.920	40.0 " 86.5	1.4703
Spearmint	0.92 " 0.98	−104.0 " −138.0	1.4702
Peppermint	0.90 " 0.925	−72.0 " −95.2	1.4559
Wintergreen	1.180 " 1.187	Inactive	1.4200
Cassia bark	1.055 " 1.065	Very slightly levo gyrate	1.5025
Mace	0.91 " 0.93	20.0 to 58.0	1.4774
Ginger	0.875 " 0.885	−75.6 " −120.9	— —
Almonds (bitter)	1.045 " 1.06	Inactive	1.5373

With the exception of oil of orange, these oils vary so widely in their optical rotation as to be incapable of being estimated in extracts by the polarimetric method described for lemon oil.

ORANGE EXTRACT.

Orange oil is a yellowish liquid, having the characteristic odor of orange, and a mild aromatic taste. It is prepared from orange peel in an analogous manner to that of lemon oil, which it somewhat resembles in chemical composition. At least 90% of orange oil, according to Walach, consists of dextro-limonene (citrene). It has a much higher specific rotary power than lemon oil. For the determination of oil in orange extract, follow the method given for oil in lemon extract, page 742, dividing the direct polarimetric reading on the Ventzke scale, calculated for the 200-mm. tube, by 5.3 to obtain the per cent of orange oil, by volume. To obtain the per cent by weight, multiply the per cent by volume by 0.85 and divide by the specific gravity of the extract.

ALMOND EXTRACT.

Essence of bitter almonds, or *Spiritus amygdalæ amaræ*, is thus prepared according to the U. S. Pharmacopœia:

Oil of bitter almonds	10 cc.
Alcohol.	800 cc.
Distilled water sufficient to make.	1000 cc.

Thus 1% of almond oil is present in the product.

Oil of bitter almonds is obtained by subjecting crushed bitter almonds or apricot seeds to distillation with water. It should be remembered that both sweet and bitter almonds yield a bland fixed oil on pressure, which is not to be confounded with the volatile oil yielded on distillation of the bitter almonds after the fixed oil has been pressed out. Bitter



Benzaldehyde or purified oil of bitter almonds has a yellowish color, a bitter, acrid, burning taste, and a marked almond odor. The specific gravity of the crude oil varies from 1.052 to 1.082, while that of the purified oil (benzaldehyde) at 20° is 1.0455. Its boiling-point is 180° C. On standing it becomes readily oxidized to benzoic acid. It is readily soluble in alcohol and ether. Its solubility in water is slight, 1:300. Its index of refraction at 20° C. is 1.5446. It should be noted that the refractive indices of almond oil, whether with or without hydrocyanic acid, and of artificial benzaldehyde are nearly the same.

Benzaldehyde is produced artificially in a variety of ways, but is chiefly prepared by the action of chlorine on hot toluene. The resulting benzyl chloride is distilled with lead nitrate and water in an atmosphere of carbon dioxide, which forms benzoic aldehyde. Synthetic benzaldehyde has the same properties as the purified oil of bitter almonds, and has largely displaced it in the market, not the least of its advantages being its freedom from hydrocyanic acid.

Adulteration of Almond Oil.—The official essence of the Pharmacopœia does not specify that the almond oil used be perfectly free from hydrocyanic acid, in spite of the fact that its highly poisonous nature is well known, and that it exists in the crude oil to the extent of from 4 to 6 per cent. True, but little of it is found in the extract, but in these days, when the unannounced presence in foods of such substances as antiseptics and coloring matters is regarded as questionable from a sanitary standpoint, in spite of the fact that their toxic effects on man are still matters of controversy, there should be little hesitancy in pronouncing the presence of prussic acid objectionable, especially when a pure almond oil entirely free from it is readily obtainable.

The presence of nitrobenzol or oil of mirbane as a substitute of almond oil is to be looked for. This substance is sometimes, though incorrectly, called artificial oil of bitter almonds. It is a heavy, yellow liquid of the composition $C_6H_5NO_2$, readily soluble in water. Its specific gravity at 20° C. is 1.2039. Its boiling-point is 205° C. It is formed by the action of nitric acid on benzol. It possesses a highly pungent odor, somewhat like that of oil of bitter almonds, though more penetrating and less refined. Its index of refraction at 20° C. is 1.5517.

Detection of Nitrobenzol.*—Boil 15 cc. of the extract in a test-tube with a few drops of a strong solution of potassium hydroxide. Nitrobenzol produces a blood-red coloration.

* Holde, Jour. Soc. Chem. Ind., 13 (1893), p. 906.

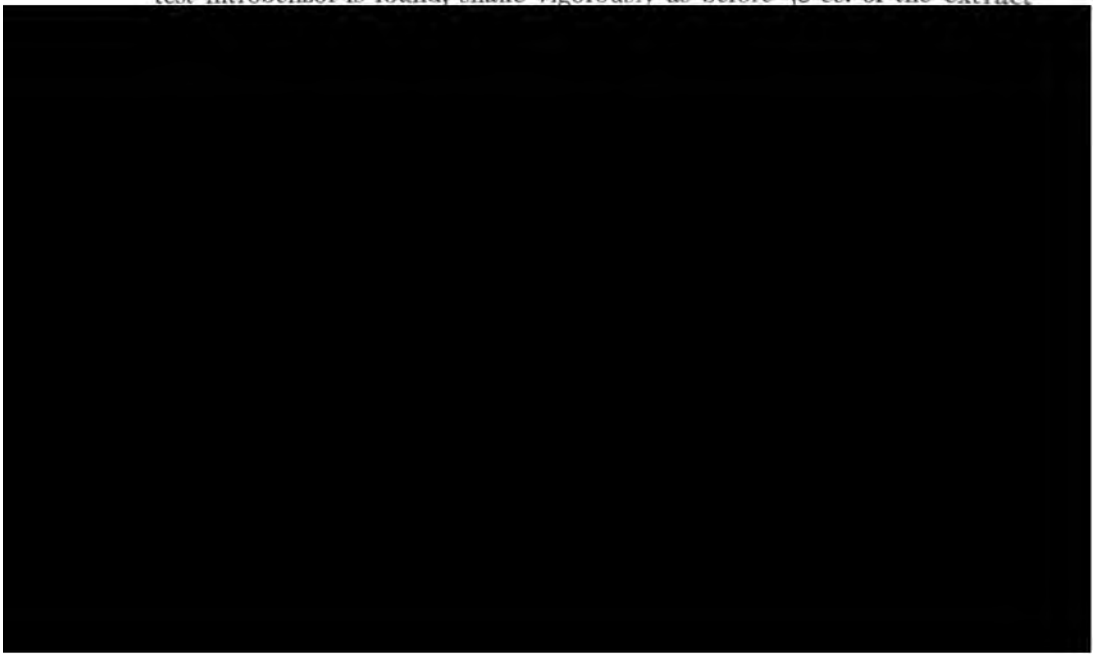
Distinction between Benzaldehyde and Nitrobenzol.—Treat 20 cc. of the extract with 5 to 10 cc. of a cold, saturated aqueous solution of sodium bisulphite in a test-tube, and shake vigorously. Transfer to an evaporating-dish, and heat on the water-bath till the alcohol is driven off. At this stage benzaldehyde remains in the hot solution as a crystalline salt, and the solution gives off no almond odor.

Nitrobenzol, on the contrary, does not combine with the bisulphite and is insoluble, forming globules of oil on the surface of the hot liquid, and in addition giving off the pungent odor so characteristic of the substance.

Determination of Benzaldehyde.—In case nitrobenzol by the qualitative test is found to be absent, shake vigorously 50 cc. of the extract with 20 cc. of the saturated sodium bisulphite solution in a stoppered flask, transfer to an evaporating-dish, and heat on the water-bath till the alcohol has disappeared, keeping up the original volume by the occasional addition of water. Note the odor of the solution at frequent intervals during evaporation, and if at any time the least odor of escaping benzaldehyde is apparent, stir in at once a few drops more of the bisulphite solution. Cool, dilute with water slightly, make strongly alkaline with sodium hydroxide, and extract in a separatory funnel with four portions of low-boiling petroleum ether of 15 to 20 cc. each. Wash the combined petroleum ether twice with water, and, after removal of the water, transfer it to a tared dish, and allow it to evaporate spontaneously at room temperature. Finally weigh the residue.

By reason of the volatility of benzaldehyde and its tendency to oxidize to benzoic acid, the results are only approximate.

Separation of Nitrobenzol and Benzaldehyde.—If by the qualitative test nitrobenzol is found, shake vigorously as before 50 cc. of the extract



separatory funnel strongly alkaline with sodium hydroxide, and shake out the benzaldehyde, if present, with petroleum ether as previously described. If after making the solution alkaline no odor of benzaldehyde is apparent, the absence of benzaldehyde may be inferred.

Distinction between Artificial Benzaldehyde and Pure Almond Oil.—Test the final residue from the ether extract by shaking with an equal volume of concentrated sulphuric acid in a test-tube. With natural oil of almonds a clear, brilliant, but dark currant-red color is produced, while with artificial benzaldehyde, the acid produces a dirty brown color with the formation of a precipitate.

Determination of Alcohol.—In the absence of other flavoring substances than nitrobenzol and benzaldehyde, which are rarely present to an extent exceeding 1%, a sufficiently close approximation for most purposes can be gained by estimating the alcohol from the direct specific gravity of the extract.

Detection of Hydrocyanic Acid.—To a few cubic centimeters of extract in a test-tube add a few drops of a mixture of solutions of ferrous sulphate and ferric chloride, the ferrous salt being in excess. Make alkaline with sodium hydroxide, and add enough dilute hydrochloric acid to dissolve the precipitate formed by the alkali. Presence of a blue coloration or precipitate, due to the formation of Prussian blue, indicates hydrocyanic acid. The reaction is very delicate.

Determination of Hydrocyanic Acid.*—Hydrocyanic acid may be determined by titration with tenth-normal silver nitrate solution. 25 cc. of the extract are measured into a flask, and 5 cc. of freshly prepared magnesium hydroxide suspended in water are added, or enough to make the reaction alkaline.

A few drops of a solution of potassium chromate are then introduced, and the tenth-normal silver nitrate solution added till, with shaking, the formation of the red silver chromate indicates the end-point. 1 cc. of silver solution equals 0.0027 gram of hydrocyanic acid.

ARTIFICIAL FRUIT ESSENCES.

Nearly all the fruits possess distinctive flavors, which are desirable in food preparations, and which may be made to impart their flavor to such substances as confections, ice cream, dessert mixtures, jellies, etc., by simply mixing with these foods the fresh or preserved fruit or fruit juice in sufficient quantity. In many cases, however, it is not found

* Vielhaber, Arch. Pharm. (3), 13, 408.

possible or practicable to prepare from the fruits themselves an extract sufficiently concentrated to give the distinctive fruit flavor, when used in moderate quantity, and hence the use of artificial fruit essences made up of compound ethers, mixed in varying combinations and proportions to imitate more or less closely various fruit flavors.

These ethers are usually much more pungent and penetrating than the fruits which they imitate, and, while lacking the delicacy and refinement of the original fruits, serve to impart a certain semblance of the genuine flavor in a convenient and highly concentrated form.

Some of the single compound ethers possess a remarkable resemblance to particular fruits, while to imitate other fruits a mixture of various ethers and flavoring materials, such as lemon and other volatile oils, vanilla, organic acids, chloroform, etc., is necessary. These artificial essences should in all cases be sold as such, and not as "pure fruit flavors."

Artificial Pineapple Essence is made up by dissolving in alcohol butyric ether, $C_4H_7(C_2H_5)O_2$, which possesses a distinct pineapple flavor, and is prepared by mixing 100 parts of butyric acid ($C_4H_8O_2$), 100 parts of alcohol, and 50 parts of sulphuric acid, and shaking. Butyric ether is sparingly soluble in water, and boils at 121° C.

Artificial Quince Essence depends as a basis on ethyl pelargonate, sometimes called pelargonic or œnanthic ether, $C_2H_5,C_9H_{17}O_2$, dissolved in alcohol. Pelargonic ether is formed by digestion with the aid of heat of pelargonic acid and alcohol. Pelargonic acid, $C_9H_{18}O_2$, is first obtained by the action of nitric acid on oil of rue. Pelargonic ether is a colorless liquid, having a specific gravity of 0.8635 at 17.5° C. Its boiling-point is 227° to 228° C. It is insoluble in water.

Artificial Jargonelle Pear Essence consists of an alcoholic solution of amyl or pentyl acetate, $C_5H_{11}C_2H_3O_2$. This is prepared by distilling

of a large variety of these artificial essences. The numerals in the various columns indicate the parts by volume to be added to one hundred parts of deodorized alcohol.

	Chloroform.	Nitrous Ether.	Aldehyde.	Acetic Ether.	Formic Ether.	Butyric Ether.	Valerianic Ether.	Benzoic Ether.	Œnanthylic Ether.	Oil of Persicot.	Sebacic Ether.	Methyl-salicylic Ether.
Pineapple	1	.....	1	.....	.....	5						
Melon	.....	.....	2	.....	1	4	5	.....	.....	.....	10	
Strawberry	.....	1	.....	5	1	5	.....	.....	.....	.....	.....	1
Raspberry	.....	1	1	5	1	1	.....	1	1	.....	1	1
Gooseberry	.....	.....	1	5	.....	.....	.....	1	1			
Grape	2	.....	2	.....	2	.....	.....	.....	10	.....	.....	1
Apple	1	1	2	1								
Orange	2	.....	2	5	1	1	.....	1	.....	.....	.....	1
Pear	.....	.....	.....	5								
Lemon	1	1	2	10								
Black cherry	.....	.....	.....	10	.....	.....	.....	5	.....	2		
Cherry	.....	.....	.....	5	.....	.....	.....	5	1			
Plum	.....	.....	5	5	1	2	.....	.....	.....	4		
Apricot	1	.....	.....	.....	.....	10	5	.....	1			
Peach	.....	.....	2	5	5	5	5	.....	.....	5	1	
Currant	.....	.....	1	5	.....	.....	.....	1	1			

	Amyl Alcohol.	Amyl-acetic Ether.	Amyl-butyric Ether.	Amyl-valerianic Ether.	Oil of Lemon.	Oil of Orange.	Saturated Alcholic Solutions of				Glycerin.
							Tartaric Acid.	Oxalic Acid.	Succinic Acid.	Benzoic Acid.	
Pineapple	.....	.....	10	.....	.....	.....	.....	.....	.....	.....	3
Melon	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	3
Strawberry	.....	3	2	.....	.....	.....	.....	.....	.....	.....	2
Raspberry	.....	1	1	.....	.....	.....	5	.....	1	.....	4
Gooseberry	.....	.....	.....	.....	.....	.....	5	.....	1	1	
Grape	.....	.....	.....	.....	.....	.....	5	.....	3	.....	10
Apple	.....	.....	.....	10	.....	.....	.....	1	.....	.....	4
Orange	.....	1	.....	.....	.....	10	1	.....	.....	.....	10
Pear	.....	2	.....	.....	.....	.....	.....	.....	.....	.....	2
Lemon	.....	.....	.....	.....	10	.....	10	.....	1	.....	5
Black cherry	.....	.....	.....	.....	.....	.....	.....	1	.....	2	
Cherry	.....	.....	.....	.....	.....	.....	.....	.....	.....	1	3
Plum	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	3
Apricot	2	.....	1	.....	.....	.....	1	.....	.....	.....	4
Peach	2	.....	.....	.....	.....	.....	.....	.....	.....	.....	5
Currant	.....	.....	.....	.....	.....	.....	5	.....	1	1	

REFERENCES ON CANNED FOODS, FRUIT PRODUCTS, AND FLAVORING EXTRACTS.

ADAMS, M. A. Composition and Adulteration of Fruit Jams. Analyst, 9, 1884, p. 100.

ANGELL, A. Microscopical Structure of Fruits, etc., to be met with in Jams and Preserves. Analyst, 1, 1877, p. 73.

BIOLETTI, F. T., and DAL PIAZ, A. M. Preservation of Unfermented Grape Must. Cal. Exp. Sta. Bul. 130.

BODMER, R., and MOOR, C. G. On Copper in Peas. Analyst, 22, 1897, p. 141.

BOSELEY, L. R. The Analysis of Marmalade. Analyst, 23, 1898, p. 123.

BROWNE, C. A. A Chemical Study of the Apple and Its Products. Penn. Dept. of Agriculture Bul. 58.

BUDDEN, E. R., and HARDY, H. Colorimetric Estimation of Lead, Copper, Tin, and Iron. Analyst, 19, 1894, 168.

DOREMUS, C. A. Collecting and Analyzing Gases in Canned Goods. Jour. Am. Chem. Soc., 19, 733.

HAUSNER, A. The Manufacture of Preserved Foods and Sweetmeats. London, 1902.

HESS, W. H. The Distinction of True Extract of Vanilla from Liquid Preparations of Vanillin. Jour. Am. Chem. Soc., 21, 1899, p. 719.

HESS, W. H., and PRESCOTT, A. B. Coumarin and Vanillin, their Separation, Estimation and Identification in Commercial Flavoring Extracts. Jour. Am. Chem. Soc., 21, 1899, p. 256.

HILGARD, E. W., and COLBY, G. E. Investigations of Canned Products. Rep. Cal. Exp. Sta., 1897–98, p. 159.

HILGAR, A., u. LABAND, L. Ueber electrolytische Abscheidung von Kupfer, Zink und Zinn aus Konserven. Zeits. f. Unters. der Nahr. u. Genuss., 2, 795.

HUSMANN, G. C. Manufacture and Preservation of Unfermented Grape Must. U. S. Dept. of Agric., Bur. of Plant Ind., Bul. 24.

LADD, E. F. Food Products and their Adulteration. (Canned Goods, Ketchups, Jellies, Jams and Extracts.) North Dak. Exp. Sta., Buls. 53 and 57.

MACFARLANE, T. Unfermented Grape Juice. Can. Inl. Rev. Dept. Bul. 82.

McELROY, K. P., and BIGELOW, W. D. Canned Vegetables. U. S. Dept. of Agric.,



PRESCOTT, S. C., and UNDERWOOD, W. L. Micro-organisms and Sterilizing Processes in the Canning Industry. Tech. Quarterly, 10, 1897, p. 183; also 11, 1898, p. 6.

SPAETH, E. On Fruit Juices and their Examination, with Particular Reference to Raspberry Juice. Zeits. f. Unters. der Nahr. u. Genuss., 2, 1899, p. 633; Abs. Analyst, 25, 1900, p. 10.

TOLMAN, L. M. The Polarization of Fruits, Jellies, Jams and Honies. Jour. Am. Chem. Soc., 24, 1902, 515.

TOLMAN, L. M., MUNSON, L. S., and BIGELOW, W. D. The Composition of Jellies and Jams. Jour. Am. Chem. Soc., 23, 1901, p. 347.

TRAPHAGEN, F. W., and BURKE, E. Occurrence of Salicylic Acid in Fruits. Jour. Am. Chem. Soc., 25, 1903, p. 242.

WILSON, H. M. Adulteration of Tinned Peas by Copper. Pub. Health, Apr., 1892, p. 203.

WINTON, A. L. Detection of Coal Tar Dyes in Fruit Products. Jour. Am. Chem. Soc. 22, 1900, p. 582.

—— Beiträge zur Anatomie des Beerenobstes. Zeits. f. Unters. der Nahr. u. Genuss., 5, 1902, p. 785.

WINTON, A. L., and SILVERMAN, M. The Analysis of Vanilla Extract. Jour. Am. Chem. Soc., 24, 1902, p. 1129.

WITHERS, W. A., and PRIMROSE, H. W. Preservatives in Canned Foods in North Carolina. North Car. Exp. Sta. Bul. 165.



APPENDIX.

THE ZEISS IMMERSION REFRACTOMETER.

Several forms of refractometer have heretofore been described, all of which have been of main use in connection with the study of oils and fats. A more recently devised instrument, the immersion refractometer, made by Zeiss, has many features that especially commend it to the use of the food analyst. The construction of the immersion refractometer is such that, as its name implies, it may be immersed directly in an almost endless variety of solutions, the strength of which, within limits, may be determined by the degree of refraction read upon an arbitrary scale. Thus, for example, the strengths of various acids and of a variety of salt solutions used as reagents in the laboratory, as well as of formaldehyde, of sugars in solution, and of alcohol, are all capable of determination by the use of the immersion refractometer.

Figure 120 shows the form used by the writer. *P* is a glass prism fixed in the lower end of the tube of the instrument, while at the top of the tube is the ocular *Oc*, and just below this on a level with the vernier screw *Z* is the scale on which is read the degree of refraction of the liquid in which the prism *P* is immersed. The tube may be held in the hand and directly dipped in the liquid to be tested, this liquid being contained in a vessel with a translucent bottom, through which the light is reflected. The preferable method of use is, however, that shown in the cut.

A is a metal bath with inlet and outlet tubes, arranged whereby water is kept at a constant level. The water is maintained at a constant temperature by means of a controller of the same type as the refractometer heater shown in Fig. 92. In the bath *A* are immersed a number of beakers, containing the solutions to be tested. *T* is a frame on which is hung the refractometer by means of the hook *H*, at just the right height to permit of the immersion of the prism *P* in the liquid in any of the beakers in the row beneath. Under this row of beakers the bottom of the tank is composed of a strip of ground glass, through which light is reflected by an adjustable pivoted mirror.

The temperature of the bath is noted by a delicate thermometer immersed therein, capable of reading to tenths of a degree.

Returning to the main refractometer-tube, *R* is a graduated ring or collar which is connected by a sleeve within the tube with a compound prism near the bottom, the construction being such that by turning the collar *R* one way or the other the chromatic aberration or dispersion of any liquid may be compensated for, and a clear-cut shadow or critical line projected cross the scale. By the graduation on the collar *R*, the degree of

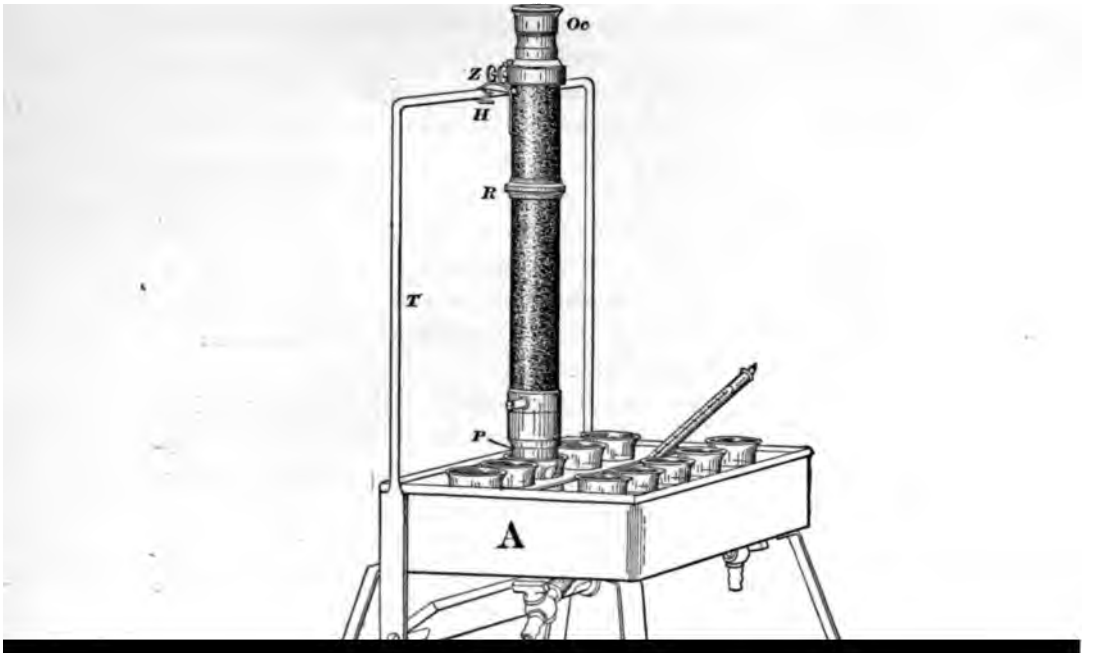


TABLE OF INDICES OF REFRACTION, n_D.

(Compared with Scale Readings of Zeiss Immersion Refractometer, according to Wagner.)

Scale Reading.	n_D.	Scale Reading.	n_D.	Scale Reading.	n_D.	Scale Reading.	n_D.	Scale Reading.	n_D.
0.0	1.327360	5.0	1.329320	10.0	1.331260	15.0	1.333200	20.0	1.335168
0.1	1.327399	5.1	1.329350	10.1	1.331299	15.1	1.333238	20.1	1.335168
2	438	2	398	2	388	2	276	2	206
3	477	3	437	3	377	3	314	3	244
4	516	4	476	4	416	4	352	4	282
5	555	5	515	5	455	5	390	5	320
6	594	6	554	6	494	6	428	6	358
7	633	7	593	7	533	7	466	7	396
8	672	8	632	8	572	8	504	8	434
9	711	9	671	9	611	9	542	9	472
1.0	750	6.0	710	11.0	650	16.0	580	21.0	510
1.1	1.327789	6.1	1.329740	11.1	1.331689	16.1	1.333619	21.1	1.335549
2	828	2	788	2	728	2	658	2	588
3	867	3	827	3	767	3	697	3	627
4	906	4	866	4	806	4	736	4	666
5	945	5	905	5	845	5	775	5	705
6	984	6	944	6	884	6	814	6	744
7	1.328023	7	982	7	932	7	833	7	783
8	062	8	1.330022	8	962	8	892	8	822
9	101	9	061	9	1.332001	9	931	9	861
2.0	140	7.0	100	12.0	040	17.0	970	22.0	900
2.1	1.328180	7.1	1.330139	12.1	1.332078	17.1	1.334008	22.1	1.335938
2	220	2	178	2	116	2	046	2	976
3	657	3	217	3	154	3	084	3	1.336014
4	300	4	256	4	192	4	122	4	052
5	340	5	295	5	230	5	160	5	090
6	380	6	334	6	268	6	198	6	128
7	420	7	373	7	304	7	236	7	166
8	460	8	412	8	344	8	274	8	204
9	500	9	451	9	382	9	312	9	242
3.0	540	8.0	490	13.0	420	18.0	350	23.0	280
3.1	1.328579	8.1	1.330528	13.1	1.332459	18.1	1.334389	23.1	1.336319
2	618	2	566	2	498	2	428	2	358
3	657	3	604	3	537	3	467	3	397
4	696	4	642	4	576	4	506	4	436
5	735	5	680	5	615	5	545	5	475
6	774	6	718	6	654	6	584	6	514
7	813	7	756	7	693	7	623	7	553
8	852	8	794	8	732	8	662	8	592
9	891	9	832	9	771	9	701	9	631
4.0	930	9.0	870	14.0	810	19.0	740	24.0	670
4.1	1.328969	9.1	1.330909	14.1	1.332849	19.1	1.334779	24.1	1.336708
2	1.329008	2	948	2	888	2	818	2	746
3	047	3	987	3	927	3	857	3	784
4	085	4	1.331026	4	966	4	896	4	822
5	125	5	104	5	1.333005	5	935	5	860
6	164	6	104	6	044	6	974	6	898
7	203	7	143	7	083	7	1.335013	7	936
8	242	8	182	8	122	8	052	8	974
9	281	9	221	9	161	9	091	9	1.337012
5.0	320	10.0	260	15.0	200	20.0	130	25.0	050

TABLE OF INDICES OF REFRACTION, n_D—(*Continued*).

Scale Reading.	n_D.	Scale Reading.	n_D.	Scale Reading.	n_D.	Scale Reading.	n_D.	Scale Reading.	n_D.
25.0	1.337050	30.0	1.338960	35.0	1.340860	40.0	1.342750	45.0	1.344630
25.1	1.337088	30.1	1.338998	35.1	1.340898	40.1	1.342788	45.1	1.344667
2	126	2	1.339036	2	936	2	826	2	704
3	164	3	074	3	974	3	864	3	741
4	202	4	112	4	1.341012	4	902	4	778
5	240	5	150	5	050	5	940	5	818
6	278	6	188	6	088	6	978	6	852
7	316	7	226	7	126	7	1.343016	7	889
8	354	8	264	8	164	8	054	8	926
9	392	9	302	9	202	9	092	9	963
26.0	430	31.0	340	36.0	240	41.0	130	46.0	1.345000
26.1	1.337468	31.1	1.339378	36.1	1.341278	41.1	1.343167	46.1	1.345037
2	506	2	416	2	316	2	204	2	074
3	544	3	454	3	354	3	241	3	111
4	582	4	492	4	392	4	278	4	148
5	620	5	530	5	430	5	315	5	185
6	658	6	568	6	468	6	352	6	222
7	696	7	606	7	506	7	389	7	259
8	734	8	644	8	544	8	426	8	296
9	772	9	682	9	582	9	463	9	333
27.0	810	32.0	720	37.0	620	42.0	500	47.0	370
27.1	1.337849	32.1	1.339758	37.1	1.341657	42.1	1.343538	47.1	1.345408
2	888	2	796	2	694	2	576	2	446
3	927	3	834	3	731	3	614	3	484
4	966	4	872	4	768	4	652	4	522
5	1.338005	5	910	5	805	5	690	5	560
6	044	6	948	6	842	6	728	6	598
7	083	7	986	7	879	7	766	7	636
8	122	8	1.340024	8	916	8	804	8	674
9	161	9	062	9	953	9	842	9	712
28.0	200	33.0	100	38.0	990	43.0	880	48.0	750
28.1	1.338238	33.1	1.340138	38.1	1.342028	43.1	1.343918	48.1	1.345787
2	276	2	176	2	066	2	956	2	824
3	314	3	214	3	104	3	994	3	861

TABLE OF INDICES OF REFRACTION, n_D—(*Continued*).

Scale Reading.	n_D.	Scale Reading.	n_D.	Scale Reading.	n_D.	Scale Reading.	n_D.	Scale Reading.	n_D.
50.0	1.346500	55.0	1.348360	60.0	1.350210	65.0	1.352050	70.0	1.353880
50.1	1.346537	55.1	1.348397	60.1	1.350247	65.1	1.352087	70.1	1.353917
2	574	2	434	2	284	2	124	2	954
3	611	3	471	3	321	3	161	3	991
4	648	4	508	4	358	4	198	4	1.354028
5	685	5	545	5	395	5	235	5	065
6	722	6	582	6	432	6	272	6	102
7	759	7	619	7	469	7	309	7	139
8	796	8	656	8	506	8	346	8	176
9	833	9	693	9	543	9	383	9	213
51.0	870	56.0	730	61.0	580	66.0	420	71.0	250
51.1	1.346907	56.1	1.348767	61.1	1.350617	66.1	1.352457	71.1	1.354286
2	944	2	804	2	654	2	494	2	322
3	981	3	841	3	691	3	531	3	358
4	1.347018	4	878	4	728	4	568	4	394
5	055	5	915	5	765	5	605	5	430
6	092	6	952	6	802	6	642	6	466
7	129	7	989	7	839	7	679	7	502
8	166	8	1.349026	8	876	8	716	8	538
9	203	9	063	9	913	9	753	9	574
52.0	240	57.0	100	62.0	950	67.0	790	72.0	610
52.1	1.347277	57.1	1.349137	62.1	1.350987	67.1	1.352827	72.1	1.354646
2	314	2	174	2	1.351024	2	864	2	682
3	351	3	211	3	061	3	901	3	718
4	388	4	248	4	098	4	938	4	754
5	425	5	285	5	135	5	975	5	790
6	462	6	312	6	172	6	1.353012	6	826
7	499	7	359	7	209	7	049	7	862
8	536	8	396	8	246	8	086	8	898
9	573	9	433	9	283	9	123	9	934
53.0	610	58.0	470	63.0	320	68.0	160	73.0	970
53.1	1.347647	58.1	1.349507	63.1	1.351357	68.1	1.353196	73.1	1.355006
2	684	2	544	2	394	2	232	2	042
3	721	3	581	3	431	3	268	3	078
4	758	4	618	4	468	4	304	4	114
5	795	5	655	5	505	5	340	5	150
6	832	6	692	6	542	6	376	6	186
7	869	7	729	7	579	7	412	7	222
8	906	8	766	8	616	8	448	8	258
9	943	9	803	9	653	9	484	9	294
54.0	980	59.0	840	64.0	690	69.0	520	74.0	330
54.1	1.348018	59.1	1.349877	64.1	1.351726	69.1	1.353556	74.1	1.355366
2	056	2	914	2	762	2	592	2	402
3	094	3	951	3	798	3	628	3	438
4	132	4	988	4	834	4	664	4	474
5	170	5	1.350025	5	870	5	700	5	510
6	208	6	062	6	906	6	736	6	546
7	246	7	099	7	942	7	772	7	582
8	284	8	136	8	978	8	808	8	618
9	322	9	173	9	1.352014	9	844	9	659
55.0	360	60.0	210	65.0	050	70.0	880	75.0	690

TABLE OF INDICES OF REFRACTION, n_D—(*Continued*).

Scale Reading.	n_D.	Scale Reading.	n_D.	Scale Reading.	n_D.	Scale Reading.	n_D.	Scale Reading.	n_D.
75.0	1.355690	80.0	1.357500	85.0	1.359300	90.0	1.361090	95.0	1.362870
75.1	1.355727	80.1	1.357536	85.1	1.359336	90.1	1.361126	95.1	1.362906
2	764	2	572	2	372	2	162	2	942
3	801	3	608	3	408	3	198	3	978
4	838	4	644	4	444	4	234	4	1.363014
5	875	5	680	5	480	5	270	5	050
6	912	6	716	6	516	6	306	6	086
7	949	7	752	7	552	7	342	7	122
8	986	8	788	8	588	8	378	8	158
9	1.356023	9	824	9	624	9	414	9	194
76.0	060	81.0	860	86.0	660	91.0	450	96.0	230
76.1	1.356096	81.1	1.357896	86.1	1.359696	91.1	1.361486	96.1	1.363256
2	132	2	932	2	732	2	522	2	292
3	168	3	968	3	768	3	558	3	328
4	204	4	1.358004	4	804	4	594	4	364
5	240	5	040	5	840	5	630	5	400
6	276	6	076	6	876	6	666	6	436
7	312	7	112	7	912	7	702	7	472
8	348	8	148	8	948	8	738	8	518
9	384	9	184	9	984	9	774	9	554
77.0	420	82.0	220	87.0	1.360020	92.0	810	97.0	590
77.1	1.356456	82.1	1.358256	87.1	1.360056	92.1	1.361846	97.1	1.363625
2	492	2	292	2	092	2	882	2	660
3	528	3	328	3	128	3	918	3	695
4	564	4	364	4	164	4	954	4	730
5	600	5	400	5	200	5	990	5	765
6	636	6	436	6	236	6	1.362026	6	800
7	672	7	472	7	272	7	062	7	835
8	708	8	508	8	308	8	098	8	870
9	744	9	544	9	344	9	134	9	905
78.0	780	83.0	580	88.0	380	93.0	170	98.0	940
78.1	1.356816	83.1	1.358616	88.1	1.360416	93.1	1.362205	98.1	1.363975
2	852	2	652	2	452	2	240	2	1.364010
3	888	3	688	3	488	3	275	3	045

SCALE READING AND INDEX OF REFRACTION OF DISTILLED WATER AT 10–30° C., ACCORDING TO WAGNER.

Temperature C.	Scale Reading.	Index of Refraction, n_D.	n_D Difference for 1° C.	Temperature C.	Scale Reading.	Index of Refraction, n_D.	n_D Difference for 1° C.
30	11.8	1.33196	. . .	19	14.7	1.333075	8.5
29	12.1	1.33208	12.0	18	14.9	1.33316	8.5
28	12.4	1.332105	11.5	17.5	15.0	1.33320	4 } 8.0
27	12.7	1.33231	11.5	17	15.1	1.33324	4 }
26	13.0	1.33242	11.0	16	15.3	1.333315	7.5
25	13.25	1.332525	10.5	15	15.5	1.33339	7.5
24	13.5	1.332625	10.0	14	15.7	1.33346	7.0
23	13.75	1.33272	9.5	13	15.85	1.333525	6.5
22	14.0	1.33281	9.0	12	16.0	1.33359	6.5
21	14.25	1.33290	9.0	11	16.15	1.33365	6.0
20	14.5	1.33299	9.0	10	16.3	1.333705	5.5

SCALE READINGS AT TEMPERATURES FROM 10–30° C.

CORRECTED TO 17.5°, ACCORDING TO WAGNER.

No.	1.	2.	3.	4.	5.	6.	7.	8.	9.	10.	11.	12 & 13.	No.
Temperature C.	Scale Reading at 17.5° C.												Temperature C.
	15.	20.	25.	30.	35.	40.	45.	50.	60.	70.	80.	90 & 100.	
30	+3.20	3.15	3.25	3.40	3.55	3.65	3.90	4.05	4.20	4.60	4.80	5.25	30
29	2.90	2.85	2.95	3.10	3.25	3.35	3.55	3.75	3.90	4.25	4.45	4.85	29
28	2.60	2.55	2.65	2.80	2.95	3.05	3.25	3.45	3.60	3.90	4.10	4.50	28
27	2.30	2.25	2.35	2.50	2.65	2.75	2.95	3.15	3.30	3.50	3.75	4.10	27
26	2.00	1.95	2.05	2.20	2.35	2.45	2.55	2.80	2.95	3.10	3.30	3.65	26
25	1.75	1.75	1.80	1.90	2.05	2.15	2.25	2.45	2.60	2.70	2.95	3.20	25
24	1.50	1.45	1.55	1.60	1.75	1.85	1.95	2.10	2.25	2.35	2.55	2.75	24
23	1.25	1.25	1.30	1.35	1.45	1.55	1.65	1.75	1.90	2.00	2.15	2.35	23
22	1.00	1.00	1.05	1.10	1.15	1.25	1.30	1.40	1.55	1.65	1.75	1.90	22
21	0.75	0.75	0.80	0.85	0.90	0.95	1.05	1.05	1.20	1.25	1.35	1.45	21
20	0.50	0.50	0.55	0.60	0.65	0.65	0.75	0.75	0.85	0.90	0.95	1.05	20
19	0.30	0.30	0.30	0.35	0.40	0.40	0.45	0.45	0.45	0.55	0.55	0.60	19
18	0.10	0.10	0.10	0.15	0.15	0.15	0.15	0.15	0.15	0.20	0.20	0.20	18
17.5	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	17.5
17	−0.10	0.10	0.10	0.10	0.10	0.10	0.15	0.15	0.15	0.15	0.20	0.20	17
16	0.30	0.30	0.30	0.30	0.35	0.35	0.40	0.45	0.45	0.50	0.55	0.55	16
15	0.50	0.45	0.45	0.50	0.60	0.60	0.65	0.75	0.75	0.80	0.85	0.90	15
14	0.70	0.60	0.60	0.70	0.80	0.85	0.90	0.95	1.05	1.10	1.25	1.25	14
13	0.85	0.75	0.75	0.85	1.00	1.10	1.15	1.20	1.35	1.40	1.55	1.60	13
12	1.00	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	12
11	1.15	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	11
10	1.25	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
No.	1.	2.	3.	4.	5.	6.	7.	8.	9.	10.	11.	12 & 13.	No.

Adjustment of the Ocular Scale.—The instrument is so adjusted that the critical line for distilled water at the temperature of 17.5° C. has a scale reading of exactly 15, corresponding to an index of refraction of 1.33300.

Strengths of Various Solutions.—The most extensive work on the quantitative determination of the strength of a large number of common aqueous solutions with the immersion refractometer has been done by Wagner, who has published a large number of tables. These tables show the percentage strength (grams per 100 cc. at 17.5° C.) of a large number of salt solutions and of acids, corresponding to the range of scale readings of the instrument, as well as of cane sugar, dextrose, formaldehyde, alcohol, etc. All these observations have been based on the Müller line, at a temperature of 17.5°. More convenient for the American analyst would be tables based on the use of a higher temperature, say

SCALE READINGS ON IMMERSION REFRACTOMETER OF VARIOUS STANDARD REAGENTS USED IN VOLUMETRIC ANALYSIS.*

	Temperature C.								
	15°.	16°.	17°.	18°.	19°.	20°.	21°.	22°.	23°.
Hydrochloric acid									
Normal	37 .45	37 .20	37 .01	36 .81	36 .59	36 .36	36 .13	35 .90	35 .66
Decinormal	0° 36	0° 16	0° 00	0° 16	0° 32	0° 48	1° 04	1° 20	1° 36
Sulphuric acid									
Normal	29 .36	29 .21	29 .06	28 .91	28 .76	28 .61	28 .46	28 .31	28 .16
Decinormal	2 .75	2 .89	3 .04	3 .19	3 .34	3 .49	3 .64	3 .79	3 .94
Decinormal	1° 38	1° 53	2° 08	2° 23	2° 38	2° 53	3° 08	3° 23	3° 38
Oxalic acid									
Normal	21 .45	21 .30	21 .15	21 .00	20 .85	20 .70	20 .55	20 .40	20 .25
Decinormal	1° 45	1° 60	1° 75	1° 90	2° 05	2° 20	2° 35	2° 50	2° 65
Tartaric acid									

[redacted]

20°, and the analyst is recommended to work out his own standards for comparison, at the temperature best suited to his special locality and convenience. The instrument is especially useful in preparing normal and tenth-normal solutions.

The table on page 764, from Wagner, shows the strength of various common laboratory reagents.

DETECTION OF ADDED WATER IN MILK.—The addition of water to milk perceptibly affects the degree of refraction of its serum, to such an extent that added water may often be detected by means of the immersion refractometer. In curdling milk-samples for examination and comparison by this method, care should be taken to observe absolutely identical conditions in all cases.

I. CONSTANTS OF MILK AND MILK SERUM. LABORATORY SAMPLES.

Determinations on Milk.						On Milk Serum.	
Total Solids, Per Cent.	Water, Per Cent.	Fat, Per Cent.	Solids not Fat, Per Cent.	Ash, Per Cent.	Specific Gravity at 15° C.	Specific Gravity at 15° C.	Immersion Refractometer Reading at 20° C.
16.45	83.55	8.20	8.25	.....	1.0255	1.0274	40.95
15.90	84.10	7.00	8.90	0.69	1.0277	1.0285	42.00
14.37	85.63	5.50	8.88	0.58	1.0282	1.0280	42.40
14.17	85.83	4.85	9.32	0.62	1.0313	1.0281	44.20
14.04	85.96	4.95	9.09	0.60	1.0303	1.0274	42.70
13.80	86.20	5.00	8.80	0.65	1.0302	1.0289	42.75
13.59	86.41	4.30	9.29	0.64	1.0321	1.0285	44.50
13.39	86.61	4.40	8.99	0.50	1.0324	1.0285	43.70
13.28	86.72	4.40	8.88	0.60	1.0299	1.0289	42.65
13.12	86.88	4.00	9.12	0.59	1.0317	1.0280	43.75
13.00	87.00	4.30	8.70	0.56	1.0310	1.0266	42.60
12.90	87.10	3.85	9.05	0.61	1.0318	1.0289	43.40
12.80	87.20	4.30	8.50	0.46	1.0304	1.0277	42.70
12.70	87.30	3.80	8.90	0.53	1.0314	1.0280	43.10
12.63	87.37	3.50	9.13	0.65	1.0323	1.0277	43.65
12.62	87.38	4.10	8.52	0.52	1.0298	1.0272	42.40
12.57	87.43	3.70	8.87	0.68	1.0317	1.0278	43.15
12.47	87.53	3.60	8.87	0.65	1.0303	1.0282	43.15
12.36	87.64	3.20	9.16	0.55	1.0327	1.0282	43.25
12.30	87.70	3.20	9.10	0.62	1.0327	1.0283	44.00
12.16	87.84	4.35	7.81	0.49	1.0275	1.0265	41.10
12.00	88.00	3.40	8.60	0.62	1.0275	1.0280	41.75
11.86	88.14	3.60	8.26	0.49	1.0306	1.0266	42.40
11.67	88.33	3.95	7.77	0.48	1.0265	1.0240	39.30
11.60	88.40	2.75	8.85	0.65	1.0320	1.0282	43.55
11.50	88.50	3.45	8.05	0.51	1.0290	1.0269	41.40
11.40	88.60	3.10	8.30	0.60	1.0297	1.0278	42.00
11.25	88.75	2.80	8.45	0.58	1.0280	1.0274	40.90
11.07	88.93	3.00	8.07	0.62	1.0290	1.0270	40.75
10.69	89.31	2.95	7.74	.....	1.0288	1.0262	39.85
10.25	89.75	3.20	6.95	0.55	1.0230	1.0223	36.40
8.34	91.66	2.20	6.14	0.38	1.0224	1.0207	34.70

The method of curdling employed in the author's laboratory is that of Woodman (p. 129), using acetic acid, and Tables I–III show the variation of specific gravity and immersion refractometer readings on milk of different composition, the refractometer readings being made at 20° C.

The table on page 765 shows analyses of milk, selected from a wide range of samples regularly collected and examined in the routine of food inspection by the Massachusetts State Board of Health.

The following table (II) shows analyses of a whole milk submitted by the author to varying degrees of watering, up to 50% of added water:

II. CONSTANTS OF MILK AND MILK SERUM. A WHOLE MILK SYSTEMATICALLY WATERED.

Determinations on Milk.							On Milk Serum.	
Added Water, Per Cent.	Total Solids, Per Cent.	Water, Per Cent.	Fat, Per Cent.	Solids not Fat, Per Cent.	Ash, Per Cent.	Specific Gravity at 15° C.	Specific Gravity at 15° C.	Immersion Refractometer Reading at 20° C.
0	12.65	87.35	4.00	8.65	0.65	1.0315	1.0287	42.40
10	11.33	88.67	3.50	7.83	0.60	1.0278	1.0260	39.75
20	10.10	89.90	3.10	7.00	0.53	1.0252	1.0230	36.90
30	8.95	91.05	2.80	6.15	0.48	1.0211	1.0200	34.10
40	7.67	92.33	2.40	5.27	0.40	1.0192	1.0167	31.10
50	6.43	93.57	2.00	4.43	0.38	1.0154	1.0140	28.45

Table III shows a centrifugally skimmed milk, systematically watered up to 50% of added water, as in Table II. It will be observed that both the specific gravity and immersion refractometer readings of the serum in Table II, agree very closely with those of the skimmed milk in Table III, in cases having a corresponding amount of added water.



A comparison of the immersion refractometer readings of the serum of milk of varying quality shows at once that the refraction of the serum is a general index to watering. A reading below 40 with the above conditions carefully observed would be suspicious of added water, though 39 might more safely be placed as a limit, below which milk could be declared fraudulently watered.

REFERENCES ON THE IMMERSION REFRACTOMETER.

ACKERMANN E. ET VON SPINDLER, O. Sur la Determination de l'Extrait de la Biere. Jour. suisse de Chim. et Pharm., 1903, No. 30.

KIONKA, H. Ueber natürliche und künstliche Mineralwasser. Balneolog. Zeit., 14, Nos. 34 u. 35.

MATTHES, H. Quantitative Bestimmungen wässeriger Lösunguen mit dem Zeiss'schen Eintauch-Refraktometer. Zeits. fur Unters. der Nahr. u. Genuss., 1902, p. 1037.

—— Ueber refraktometrische analytische Bestimmungsmethoden. Zeits fur anal. Chem., 13 (1904), Heft 2.

MATTHES H., u. MÜLLER, F. Ueber die Untersuchung des Milchserums mit dem Zeiss'schen Eintauchrefraktometer. Zeits. fur öffentl. Chem., 1903, p. 173.

WAGNER, B. Ueber quantitative Bestimmungen wässeriger Lösungen mit dem Zeiss'schen Eintauch-Refraktometer. Sondershausen, 1903.

THE DETECTION OF REFINED CANE SUGAR IN MAPLE SUGAR AND SYRUP.

Method of Julius Hortvet.*—This method depends on the principle that the volume of the precipitate, by treatment of the sugar solution or syrup under fixed conditions with alumina cream and subacetate of lead, varies with the amount of refined sugar present.

Apparatus.—(1) A tube, adapted to be carried in the shield of the centrifuge. This tube, which is 15.3 cm. in length, has a wide cylindrical portion 3 cm. in diameter, narrowed at the top to a neck 2 cm. in diameter, and at the bottom to a stem graduated in tenths to 5 cc.

(2) A holder, made of pine or white wood, of a size adapted to carry the tube in the shield of the centrifuge. The holders and tubes should be arranged in balanced pairs in the centrifuge.

Procedure.—Introduce 5 cc. of syrup or 5 grams of sugar into the tube. Add 10 cc. of water, and dissolve completely. Next add 10 drops

* Submitted for adoption among the food methods of the Association of Official Agricultural Chemists.

of alumina cream, and 1.5 cc. of lead subacetate. Shake thoroughly, and allow to stand from forty-five to sixty minutes. Place the tube in its holder in the centrifuge shield, and run six minutes. If, after the end of this time, any material adheres to the sides of the wide part of the tube, loosen with a small wire or by giving the tube a slight twist, then run the tube six additional minutes, and finally read the volume of the precipitate in the stem, estimating to 0.01 cc.

Run a blank with the above reagents in water, subtracting the blank reading from that of the precipitate. In the case of syrup, reduce to the 5-gram basis by dividing by the specific gravity of the sample. If the sugar content of the sample is known, the specific gravity can be calculated from the table on page 499. For pure maple syrup 1.33 is very nearly correct.

The following table shows results obtained by Hortvet on two samples of maple syrup of known purity, mixed with varying amounts of refined cane sugar syrup of the same density:

Purity.	Five Minutes.	Computed Precipitate.	Difference.	Ten Minutes.	Computed Precipitate.	Difference.	Twelve Minutes.	Computed Precipitate.	Difference.
Per Cent.	cc.	cc.	cc.	cc.	cc.	cc.	cc.	cc.	cc.
100	4.90			3.29			2.80		
25	1.00	1.22	0.22	0.70	0.82	0.12	0.65	0.70	0.05
50	3.60	2.45	1.15	1.50	1.54	0.14	1.40	1.40	0.00
75	4.60	3.67	0.93	2.77	2.47	0.30	1.90	2.10	0.20
100	5.40			4.40			3.75		
10	0.30	0.54	0.24	0.28	0.44	0.16	0.28	0.37	0.09
20	1.27	1.08	0.19	0.80	0.88	0.70	0.70	0.75	0.05
60	5.00	3.24	1.76	2.70	2.64	0.06	1.70	2.25	0.55

The blank at the end of twelve minutes was 0.44 cc. The machine

of similar experiments with his own centrifuge, and work out his own standards. Results may be better compared with each other, if calculated on the water-free basis.

In case of doubt, and in fact in all cases at first, it would be well to make confirmatory tests, such as determining the ash and reducing sugar.

Data on the Ash of Maple Products.—C. H. Jones, of the Vermont Experiment Station, has furnished the data found in the table on p. 770. Samples marked pure were of unquestioned authenticity, and, unless otherwise specified, were of high grade.

Pure maple products from farmers rarely run below 0.6% in total ash, while pure manufactured maple syrups are sometimes lower, due to more complete settling and separation of the sediment by filtration.

Jones places the lowest limit of ash in pure maple syrup as 0.5%, on the basis of 11 pounds of syrup to the gallon.

POLARIZATION OF COMMERCIAL GLUCOSE.

In the formula given on page 505 for determining commercial glucose in molasses and syrup, 175 has been assumed as the maximum polarization of 42° Bé. glucose, based on the previous experience of the author.

It seems to be a fact that the polarization of this grade of glucose is somewhat lower now than formerly. The analyst is therefore recommended to keep informed by experiment from time to time as to its limits of polarization, and to use the highest limit found as a factor in the formula.

Whatever factor be adopted, however, by always expressing the final result *in terms of glucose polarizing at that factor*, a definite statement is made. In the case of honey and similar products, note the correction given on page 515, to be applied to the assumed factor.

ASH DATA ON PURE AND ADULTERATED MAPLE PRODUCTS.

	Pure Maple Syrup.		Pure Maple Sugar.			Maple Sugar Adulterated with Cane Sugar.					Brown Sugar.	
	1	2	3	4	5†	6‡	7§	8‖	9	10	11¶	12**
.....	5.34	5.23	5.22	5.25	5.59	10.	5.07	5.00	5.00	5.00	5.00	5.11
.....	0.65	0.63	0.73	0.90	1.32	0.19	0.31	0.21	0.32	0.38	1.06	2.80
.....	0.46	0.44	0 50	0.60	0.57	0.14	0.19	0.13	0.19	0.18	1.00	2.15
.....	0.19	0.19	0.23	0.30	0.75	0.05	0.12	0.08	0.13	0.20	0.06	0.65
.....	5.75	5.50	5.75	7.50	13.25	3.75	3.00	2.40	2.25	3.30	2.75	6.75
.....	2.75	2.50	2.75	3.50	3.00	1.00	1.50	1.00	1.00	1.00	1.50	0.75
.....	3.00	3.00	3.00	4.00	10.25	2.75	1.50	1.40	1.25	2.30	1.25	6.00
.....	164	167.	151.	158.	179.	197.	188.	229.	141.	172.	52	47.2
.....	110.	109.	106.	110.	94.	71.	150.	154.	106.	109.	30.2	6.8
.....	300.	300.	250.	256.	244.	550.	250.	350.	189.	230.	391.	182.
.....1:	1.09	1.20	1.09	1.14	3.41	2.75	1.	1.4	1.25	2.3	0.83	8.
t of 1 gram												
.....1:	2.73	2.75	2.36	2.33	2.60	6.4	1.66	2.27	1.78	2.11	12.9	26.8

normal acid. A measured volume of $\frac{N}{10}$ HCl is added in excess, and titrated back with $\frac{N}{10}$ NaOH, using preferably

§ 67% added cane sugar.
‖ 83% " " "

¶ Very light colored (commercial).
** Very dark colored (commercial).

INDEX.

Abbé refractometer, 389, 397
Abrastol, 678
Abrus, 38
Absinthe, 605
Acetyl value, 400
Acid fuchsin, 644, 652
Acids, fatty, 371, 375, 402, 403
of acetic series, 368
of linoleic series, 369
of oleic series, 369
mineral, in vinegar, 616, 617
organic, 42
Adams' fat method, 98
"Aerated" butter, 438
Agar agar, in jelly, 724
Aging of liquors, 594, 595
Albumin, determination in milk, 110
of muscle, 165
preparation of, 205
Albuminates, 38
Albuminoids, 39
Albumins, 37, 229
Albumose, 38
Alcohol, detection, 527
determination, 528
by distillation, 528
by ebulioscope, 545
by evaporation, 530
from specific gravity, 529
extract of spices, 312
methyl-, 743
preparation of, 593
stills, 530
tables, 531–544
Alcoholic beverages, see also liquors, 523, 524
references on, 607
state control of, 524
toxic effect of, 525
fermentation, 523
Ale, 576
Aleurone, 78
Alkaloidal nitrogen, 41
Alkaloids, proof of absence of, 590
Alkanna tincture, 80
Allantoin, 231
Allihn's sugar method, 493
tables, 494
Allspice, 323
adulteration, 326
microscopical structure, 324
Allspice, standard, 326
tannin in, 326
Almond extract, 748
adulteration of, 749
alcohol in, 751
benzaldehyde in, 750
hydrocyanic acid in, 749, 751
nitro benzol in, 749
Almonds, bitter, oil of, 747, 748
Alum in baking powder, 255, 263
in bread, 248
in flour, 237
in pickles, 712
Alumina, determination of, 263
Aluminum salts in baking powder, 263
in cream of tartar, 263
Amagat and Jean's refractometer, 389
Amides, 40
in milk, 111
Amido acids, 40
Amido nitrogen determination, 64, 111
in wheat, 231
Ammonia, determination, 64
in baking powder, 265
in foods, 41
in milk, 111
Ammonium fluoride, 676
Amylodextrin, 303
Amyloid, 79, 80
Analyst, functions of, 3–4
Angostura, 605
Anilin orange in milk, 134, 135
Annatto in butter, 435, 436
in milk, 134, 135
tests for, 637
Antipeptones, 39
Antiseptics, see also preservatives
regulation of, 662
Apparatus, 18
Apple essence, artificial, 752, 753
Apple juice, 550
Apples, composition of, 215, 216, 550
Araban, 226
Arabinose, 225, 226
Arata's color method, 641
Army rations, 201
Arsenic detection and determination, 64, 65
compounds in colors, 631
Gutzeit test for, 511

Arsenic in beer, 580, 591, 592
in vinegar, 626
Marsh apparatus, 65
Artificial colors, 628
fruit essences, 751
sweeteners, 682
references on, 687
Asaprol, 678
Asbestos fiber, preparation of, 489
Ash, determination of, 55
of food, 42
Asparagin, 40, 231
Auramin, 630

Babcock fat method, 100
milk formulæ, 115
test bottles, 102
Badouin's sesame oil test, 420
Baking powders, 254
adulteration of, 256
alum, 255
phosphate, 255
tartrate, 254
Banana essence, artificial, 752, 753
Barium compounds in colors, 630
Bark as an adulterant, 330
Barley, 213, 242, 243
proteids, 218, 233
starch, 221
Basic colors, 639, 643
Beading oil, 597
Beans, 213
Beaumé and Brix scales compared, 499
Bechi's cottonseed oil test, 418
Beef, composition of, 167
cuts of, 165
stearin, microscopical structure, 455
tallow, 430
Beer, 574
acids in, 588
adulteration of, 578
aloes in, 591
analytical methods, 581
arsenic in, 580, 591, 592
ash of, 579
Beer, standards, 578
temperance-, 580
varieties of, 575
uno-, 581
weiss-, 576
wort, 581
gravity of, 581–587
Beeswax, 515
refractometer reading of, 516
Beet (color), 636
Bellier's peanut oil test, 425
Benches, 13
Bénédictine, 605
Benzaldehyde, 748, 750
artificial, 751
Benzoic acid, 673
detection of, 142, 673, 674
in milk, 142
Betaine, 40, 231
Beta-naphthol, 677
Bigelow and McElroy's cane-sugar method, 152
Bilberry (color), 636
Birotation, 469, 479
Bisulphites as preservatives, 675
Bitter almonds, oil of, 747
Biuret reaction, 37
Blackberry (color), 636
Blast pump, 17
Blue colors, 631, 658
"Blown" cans, 692
Bock beer, 576
"Boiled" butter, 438
Bombay mace, 365
Bomb calorimeter, 42
Borax, 667
Boric acid, 667
detection, 142, 668
determination, 667, 669, 670
in butter, 436
in meat, 174, 185
in milk, 142, 144
Bourbon whiskey, 595
Brandy, 597
adulteration of, 598

[redacted]

Buckwheat, 213, 339
Burgundy wine, artificial, 560
Butter, 157, 430
adulteration of, 434
analytical methods, 432
annatto in, 435, 436
ash in, 433
azo colors in, 436
boric acid in, 436
carrotin in, 435
casein in, 433
coloring in, 434
fat, composition of, 431
standard, 433
foam test, 446
formaldehdye in, 437
glucose in, 450
microscopical examination of, 448
milk test, 446
preservatives in, 436
references on, 458
renovated, 438
salicylic acid in, 437
salt in, 433
standard, 433
sulphurous acid in, 438
Waterhouse test, 446
Butterine, 438
Butterine oil, 423
Butyro-refractometer, 390, 443
critical line of, 443
limits of butter readings, 444
oil readings on, 398
olive and cottonseed oil readings, 415
special thermometer for, 445

Caffeine, 280
determination of, 281
in cocoa, 303
Caffeol, 289
Caffetannic acid, 289
Cake, 240
Calcium carbonate crystals, 78
oxalate crystals, 78
sucrate, 156
California wines, 558
Calorie, 42
Calorimeter, bomb, 42
oil, 387, 388
respiration, 2
Camera, 84
Cammembert cheese, 158
Canada balsam, 74
Candy, see confectionery
standard, 517
Cane sugar, 461
analysis of, 480
ash of, 463
composition of, 464
detection of, 480
in milk, 145
determination of,
by copper reduction, 497
Cane sugar, determination of, by polarimetry, 481, 508
in cereals, 227
inversion of, 483, 484
manufacture of, 463
moisture in, 481
quotient of purity, 481
refining, 466
test for, 480
Canned food, 689
analysis of, 691
antiseptics in, 705, 706
composition of, 691
decomposition of, 692
impurities in, 692
metallic impurities in, 694
method of canning, 690
references on, 754
Canned fruits, 689, 691
meats, 175
vegetables, 689, 691
Cans, detection of spoiled, 692
gases from spoiled, 693
Capers, 711
Capsicin, 342
Caramel, 638
in distilled liquors, 603
in milk, 134, 136
in vanilla extract, 739
in vinegar, 625
Carbohydrates, 41, 42, 219
of cereals, 218, 227
of eggs, 205
Carbon dioxide
in baking chemicals, 259, 260
in beer, 589
in yeast, 250
Carnin, 165
Carrot (color), 637
Casein, 38, 89
determination in milk, 209
Caseose, 38
in cheese, 161
in milk, 111
Cassia, 326
adulteration of, 330
buds, 327
microscopical structure, 318
oil, 327, 747
standard, 330
Cayenne, 341
adulteration of, 343
coal-tar colors in, 345
colors in, 345
microscopical structure, 343
mineral adulterants in, 344
oil of, 342
redwood in, 345
standard, 344
Cazeneuve's color scheme, 572
Cellulose, 79, 81, 218, 224
Centrifuge, milk-fat, 101
universal, 23

Cereals, 212
ash of, 223, 234
breakfast foods, 267, 268
cane sugar in, 227
carbohydrates of, 218, 219
separation of, 227
composition of, 212
crude fiber in, 218, 228
dextrin in, 227
hemicelluloses in, 228
methods of proximate analysis, 217
pentosans in, 228
proteids of, 228
references on, 273
starch determination in, 228
Champagne, 557
Chaptalizing, 561
Chartreuse, 605
Cheddar cheese, 158
Cheese, 157
adulteration of, 158
amides in, 161
ammonia in, 161
ash in, 159
composition of, 157
cream, 158
lactic acid in, 161
fat in, 159, 162
filled, 159
milk sugar in, 162
nitrogen, water soluble in, 161
paranuclein in, 161
peptones in, 161
proteids in, 160
sampling, 159
skimmed milk, 162
standards, 158
varieties of, 157
water in, 159
whole milk, 158
Chicory, 294, 296
Chili sauce, 709
Chilton cheese, 158
Chiretta, 591
Chlor iodide of zinc, 70
Cinnamon, microscopical structure of, 329
standard, 330
Citral, 745, 746
Citric acid in milk, 91
in fruit products, 723
in lime juice, 727
Citronellal, 746
Citronella oil, 745, 746
Clams, 200
Claret wine, 557
Clarifying reagents
in microscopy, 80
in sugar analysis, 481, 502
Clerget's formula, 483
Cloves, 314
adulteration of, 320
cocoanut shells in, 321
exhausted, 320
microscopical structure, 318
oil of, 747
standard, 320
stems, 319
tannin in, 317
Coal-tar colors, 639
acid, 639, 640, 643
Arata's method, 641
basic, 639, 640, 643
classification, 639, 644, 645
detection of, 640
double dyeing method, 641
dyeing wool by, 640
extraction by amyl alcohol, 642, 643
identification of, 650, 652
in milk, 137
in sausages, 190
Rota's scheme for, 644
separation with ether, 643
Cochineal, 638
in sausages, 190
Cocoa, 299, 300
adulteration of, 304
albuminoids in, 304
alkali in, 305
analytical methods, 302

Coffee, ash of, 290
caffeine in, 291
caffetannic acid in, 289, 291
caffeol in, 289
chicory in, 294, 296
coloring of, 298
essential oil of, 289
fat in, 289, 291
glazing of, 298
microscopical structure, 293
"pellets," 292
references on, 308
starch in, 293
substitutes, 298
Cognac, 507
oil, 508
Collagen, 40, 165
Collodion silk, 572
Colors, artificial, 628
acid fuchsin, 644
arsenic compounds, 631
barium compounds, 630
basic, 643
blue, 631, 632, 634, 658
brown, 632, 634, 656
caramel, 638
chromate of lead, 638
coal tar, 639
cochineal, 638
copper compounds, 630
cudbear, 637
extraction of, by immiscible solvents, 642
fuchsin, 643
green, 631, 632, 633, 658
harmless, 630, 632
identification of, 650, 652
indigo, 638
injurious, 630, 631
in butter, 434
in cayenne, 345
in confectionery, 518, 634
in jams and jellies, 723
in ketchup, 709
in milk, 134, 137
in mustard, 300
in sugar, 466
lead chromate, 638
lead compounds, 630
logwood, 637
mercury compounds, 631
mineral, 638
non-injurious, 630, 632
orange, 632, 633, 636, 654
orchil, 637, 642
Prussian blue, 638
reagents for identifying, 651
red, 631, 632, 636, 652
references on, 660
Rota's scheme for, 644
separation by solvents, 644
toxic effect of, 629
turmeric, 637
Colors, ultramarine blue, 638
vegetable, 635, 642
violet, 632, 634, 658
wool dyeing, 640, 642
yellow, 631, 633, 636, 637, 654
Colostrum, 93
Commercial glucose. See Glucose
"Compound" foods, 717
Compressed yeast, 250
Conalbumin, 204
Concentrated foods, 201
Condensed milk, 146
analysis of, 148
as a milk adulterant, 146
ash of, 149, 152
cane sugar in, 151, 152
composition of, 147
fat in, 149, 151, 152
milk sugar in, 150
proteids in, 150, 152
solids of, 148
standards for, 148
Confectionery, 517
alcohol in, 521
analysis of, 518
arsenic in, 521
cane sugar in, 520
colors in, 518, 521
dextrin in, 520
glucose in, 521
invert sugar in, 520
lead chromate in, 519
mineral adulterants, 519
paraffin in, 519
starch in, 520
Congluten, 38
Connective tissue, 165
Copper salts, 699
determination of, 702, 703, 704
in vinegar, 626
Copra oil, 429
Cordials, 605
analysis of, 606
composition of, 606
Corky tissue, 77
Corn, 213, 244, 245, 422
bleaching of canned, 706
oil, 422
sitosterol in, 423
proteids of, 218, 233
starch, 221
syrup, 170
Corning of meat, 173
Cottonseed, 417
oil, 417
tests for, 418
stearin, 418
Cotton's cane sugar method, 145
Coumarin, 732
determination, 737
microscopical structure, 738
Cream, 153
adulteration of, 155

Cream, analytical methods, **154**
cheese, 158
evaporated, 146, 155
gelatin in, 155
standards for, 155
sucrate of lime in, 155
test scale, 152
viscogen in, 156
Cream of tartar, 257
in wine, 579
Creatin, 165
Creatinin, 165
Crême de menthe, 606
Crême de Noyau, 605
Crude fiber, 218
in cereals, 228
Crustaceans, 200
Crystals, plant, 78
Cucumber pickles, 711
Cudbear, 637
Cuprammonia, 81
Curaçoa, 605
Curcuma, 350
Curcumin, 351
Curd tests in butter, 747, 750
Curing meat, 173
Currant (color), 636
Curry powder, 350
Custard powders, 210

Dakota mustard, 360
Date stones, 297
Defren-O'Sullivan sugar
method, 114, 489
Defren's sugar tables, 490
Desiccated egg, 209
Deutyro-albumose, 30
Dextrin, 470
determination of,
in cereals, 228
in glucose, 510
in honey and molasses, 506
Dextrose, 468
determination of, 486, 487, 508
Diastase in malt extract, 592, 593

Ebulioscope, 545
Edam cheese, 187
Edestin, 231
Eggs, 203
analytical methods, 207
ash of, 206
carbohydrates of, 205
composition of, 203
desiccated, 209
fat of, 205, 206
lecithin determination, 207
physical examination of, 209
preservation of, 208
proteids of, 205
references on, 211
substitutes for, 209
waterglass as a preservative, 208
weights of, 206
white of, 204
yolk of, 205
Elaïdin oil test, 402
Elastin, 40, 165
Elderberry (color), 636
Electrolytic apparatus, 493
Elm bark, 330
Emergency rations, 201
Ergot, 239
Essential oils, 745, 747
Ether, ethyl, preparation of absolute, 58
petroleum, preparation of, for a solvent, 58
Eucasin, 122
Eugenol, 314
Ewe's milk, 91
Exhausted cloves, 320
ginger, 347
tea leaves, 285
vanilla beans, 731
Exhaust pump, 18
Extraction with immiscible solvents, 60
volatile solvents, 58

"Faints," 595
Farinaceous infants' foods, 270
Fat globules, 78



Fehling's solution, 485
equivalents of, 487
Fermentation, acetic, 609
alcoholic, 523
lactic, 93
proteolytic, 122, 157
Fermented liquors, 548
Feser's lactoscope, 127
Fibrin, 89
Fibro vascular tissue, 76
Filled cheese, 159
Fish, composition of, 198
preservatives in, 201
Flavoring extracts, 728, 747
references on, 754
Flesh bases, 165, 184, 192
foods, 165
references on, 202
Floor, 13.
Flour, 235, 241
adulteration of, 237
alum in, 237
cold water extract of, 238
gluten in, 238
inspection, 236
Fluoborates, 676, 677
Fluorides, 676
Fluosilicates, 676, 677
"Foam" test for butter, 446
Food adulteration, 5
analysis from dietetic standpoint, 2
analysis, general methods, 4
references on, 67
concentrated, 201
economy, references on, 44
inspection, 3, 5, 6, 9
references on, 10
nature and composition of, 35
standards, 3
state control of, 1
references on, 10
Fore milk, 92
Foreshots, 595
Formaldehyde, 138, 144, 664
detection of, 140, 666
determination of, 141, 665, 667
resorcin test for, 666
Fortified wine, 555
Frozen milk, test for, 93
meat, 191
Fruit, 216
candied, 519
composition of, 215
essences, artificial, 751, 753
juices, 714, 715, 725, 726
methods of proximate analysis, 217
products, 689
references on, 754
references on, 273
sugar. See Levulose.
sugar-coated, 519
sugar in, 462
Fruit, syrups, 728
tissues under the mi
Fuchsin, 643, 648
Fuel value, 42
Funnel, jacketted, 371
Furfurol, 225, 421
in distilled liquors, 605
in vinegar, 625
Fusel oil, 593, 594
detection, 601
determination, 601
toxic effect of, 597
Fustic, 636

Game, composition of, 170
Gases, in spoiled cans, 693
Geissler's carbon dioxide apparatus, 435
Gelatin, 40, 165
in cream, 155
in meat, 183
Gerber's milk centrifuge, 100
Gill and Hatch's oil calorimeter, 387
Gin, 600
Ginger, 345
adulteration of, 350
black, 346
cold water extract of, 348
exhausted, 347
liming of, 346
microscopical structure of, 349
oil of, 346, 747
root, 346
standard, 350
white, 346
Gliadin, 230, 232
Globulins, 37, 229
Globulose, 38
Glucin, 687
Glucose, 470
analysis of, 509
arsenic in, 511
composition of, 471
determination of, in honey, 514, 769
in molasses, 505, 769
dextrin in, 510
healthfulness of, 471
in beer, 577, 579
in butter, 450
polarization of, 769
test for, 510
Glucoses, 461
Gluten, 230, 231, 238
Gluten flour, 239
Glutenin, 230, 232
Glycerin in vanilla extract, 738
in wine, 570
Glycerin jelly, 74
Glycogen, 166
detection, 187
determination, 188
Goat's milk, 91
Graham flour, 236

Grape juice, 726
Grape sugar. See Dextrose
Grape sugar, standard, 469
Green colors, 631, 658
Groats, 235
Gruyère cheese, 158
Gums, 77
Gunning-Arnold nitrogen method, 334
Gunning nitrgoen methods, 61, 63
Gutzeit arsenic test, 511

Hæmoglobin, 165
Halphen cottonseed oil test, 419
Hanus' iodine absorption method, 383
Hefelmann's Bombay mace test, 365
Hehner and Richmond's milk formula, 115
Hehner's method for insoluble fatty acids, 379
Heidenhain's tartaric acid method, 262
Hemicellulose, 225, 228
Hemipeptones, 39
Hetero-albumose, 38
Hock wine, 559
Hoffmeister's schälchen, 58
Holstein cows, milk from, 124
Honey, 511
  adulteration of, 513
  analysis of, 512
  composition of, 511
  gelatin in, 513
  glucose in, 514, 769
  invert sugar in, 513
Hoods, 14
Hops, 575
  substitutes, 577
Horseflesh, characteristics of, 186
  composition of, 176
  detection of, 187, 189
  glycogen in, 187
Horse radish, 712
Hortvet's maple sugar method, 767
Hübl's iodine absorption method, 380
Human milk, 91, 119
Hungarian red pepper, 341
Hunt's iodine reagent, 384

Infants' foods, analytical methods, 271
  classification of, 269
  cold water extract of, 272
  microscopical examination of, 273
  preparation of, 269
Inosite, 166, 217
Inspection of foods, 3, 5, 6, 9
  flour, 536
  liquors, 525
  milk, 123
Inulin, 217
Invalids' foods, 269
Inversion, 461
Invert sugar, 484
  detection of, 484, 507
  determination of, 486, 487
Iodine absorption of oils, 380, 383, 384
Iodine in potassium iodide, 79
Irish whiskey, 595, 597

Jams, 712. See also Jellies
  adulteration of, 713, 717
  analysis of, 718
  apple stock in, 725
  composition of, 715
  dextrin in, 722
  glucose in, 721
  polarization of, 719
  starch in, 724
  sugars in, 719, 721
Jellies, 712, 713. See also Jams
  adulteration of, 713, 716
  agar agar in, 724
  analysis of, 718
  coagulator in, 716
  coloring matter in, 723
  composition of, 714
  gelatin in, 724
  glucose in, 721
  preservatives in, 724
  sugars in, 719, 720

Kephir, 122

Lactated infants' foods, 270
Lactoglobulin, 89
Lactometer, 95, 127
Lactoscope, 127
Lactose, 472
detection of, 507
determination of, 486, 488, 508
in milk, 111
Soxhlet's table for, 116
Lager beer, 575
Lamb, composition of, 169
cuts of, 169
Landwehr's glycogen method, 188
Lard, 451
adulteration of, 453
back, 451
"compound," 453
iodine number, 456
kettle rendered, 451
leaf, 452
microscopical examination of, 454
neutral, 452
oil, 452
references on, 459
standards, 453
stearin, 453
substitutes, 456
Laurent's saccharimeter, 478
Lead chromate, 519, 638
Lead, salts of, 694, 698
determination of, 701, 702, 704
Leavening materials, 249
references on, 275
Lecithin, 41
determination of, 207
Leffmann and Beam's method for volatile fatty acids, 377
milk centrifuge, 100
Legumes, 213
ash of, 234
Legumin, 38
Lemon extract, 739
adulteration of, 739
alcohol in, 742
analysis of, 741
citric acid in, 744
colors in, 744
composition of, 741
lemon oil in, 742, 744, 745
methyl alcohol in, 743
standard for, 739
tartaric acid in, 744
Lemongrass oil, 745, 746
Lemon juice, 727
Lemon oil, 745, 747
determination of, 742
Lentils, 213
Leucosin, 231
Levallois' bromine absorption method, 385
Levulose, 469
determination of, 486, 487, 508
Liebig's meat extract, 192
Lighting, 13
Lignin, 80
Lime, determination of, 264
in spices, 312
juice, 727
sucrate of, 155
water, in vinegar analysis, 615
Liming of ginger, 346
Limonene, 746
Liqueurs, 605
analysis of, 606
Liquor inspection, 525
Liquors, alcohol in, 527, 528
analytical methods, 527
ash of, 547
distilled, 593
analytical methods, 601
extract of, 547
fermented, 548
malted and non-malted, 578
preservatives in, 547
specific gravity of, 527
Lobster, composition of, 200
Logwood, 637
Long pepper, 340
Low wines, 595

Macaroni, 266
Macassar mace, 365
Mace, 360, 363
adulteration of, 364
Bombay, 365
Macassar, 365
microscopical structure of, 364
oil of, 747
standard, 364
Madeira wine, 557
Maize. See Corn
Malaga wine, 560
Malic acid in cider, 569
in vinegar, 617
in wine, 569
Mallet's phosphotungstic acid method, 182
Malt, 574
extracts, 185, 224, 592, 593
liquors, 574, 578
substitutes, 577
vinegar, 612
Malting, 574
Maltose, 469
detection of, 507
determination of, 486, 488, 508
Maple sap, 466
sugar, 466
adulteration of, 467, 767
ash of, 467, 769, 770
syrup, 466
adulteration of, 467, 767
ash of, 467, 769, 770
Maraschino, 605
Mare's milk, 91
Marigold, 637
Marpmann's color method, 191
Marsh arsenic apparatus, 65

Martin's color scheme, 434
"Materna" milk modifier, 121
Maumené thermal test, 386
Mayrhofer's glycogen method, 189
McGill's drying oven, 481
Meal, microscopical examination of, 239
Meat, 165
antiseptics in, 174
bases, 165, 184, 192
boric acid in, 174, 185
canned, 176
canning of, 175
colors in, 190
composition of, 165, 176
cooking, effect of, 174
corning of, 173
curing of, 173
extracts, 192, 194
albumin, acid, 197
coagulable, 197
albumoses, 197
amido nitrogen, 196
ash in, 195
fat in, 195
flesh bases in, 196, 197
meat fiber in, 197
nitrogen compounds of, 196
peptones in, 197
preservatives in, 198
proteoses in, 197
fat, composition of, 179
fiber determination, 181
gelatin determination, 183
inspection, 171
manufactured, 172
nitrates in, 184
nitrogen determination, 180–182
nitrogenous bodies, separation of, 180
peptones in, 165, 181
phosphotungstic acid method, 182
pickled, 172
prepared, 179
preservation of, 172
preservatives in, 174, 184
proteoses in, 165, 181
Microscope, reagents for, 78
stand, 70
Microscopical diagnosis, 74
Milk, 88
Milk, acidity of, 117
adulteration of, 123, 124
anilin orange in, 134, 135
annatto in, 134, 135
ash of, 91, 98
ashing of, 133
ass's, 91
boiled milk, detection, 119
boric acid in, 142
caramel in, 134, 136
carbonate in, 142
citric acid in, 91
coloring matter in, 134–137
composition of, 88–90
ewe's, 91
fat of, 91, 98
fermentations of, 93
foods, prepared, 121
fore milk, 92
formaldehyde in, 138, 140
goat's, 91
human, 91, 119
inspection, 123
known purity, 124, 125
mare's, 91
microscopical appearance, 88
modified, 119
nitrogen compounds in, 89
powder, 121
preservatives in, 137, 143
proteids of, 89
records of analysis of, 132
references on, 163
ropy, 94
sampler, 95
serum, refraction of, 129, 765, 767
specific gravity of, 129
skimmed, 125
sour, analysis of, 146
souring of, 93
standards, 124, 126

Moisture, determination of, 55
Molasses, 463, 464
adulteration of, 504
analysis of, 498
ashing of, 506
clarifying, 502
glucose in, 505, 769
standard for, 506, 507
sucrose in, 498
tin in, 507
vinegar, 613
Mollusks, 200
Mucin, 40
Mucoid proteid, 91
Muscle albumin, 165
fibers in meat, 165
sugar, 166, 190
Muscovado, 464
Mustard, 353
adulteration of, 359
ash of, 357
black, 353
cake, 355
coloring matter in, 360
Dakota, 360
flour, 354
microscopical structure of, 358
oil, fixed, 354, 426
volatile, 353, 357
pickles, 711
sinalbin in, 354
mustard oil, 354
sinapin sulphocyanate, 357
standard, 359
starch in, 359
turmeric in, 360
volatile oil of, 353
wheat in, 359
white, 353
Mutton, composition of, 169
cuts of, 169
tallow, 430
Myosin, 37, 149

Natural wine, 555
Neufchâtel cheese, 158
Nickel salts, 701
determination of, 705
Niebel's glycogen method, 188
Nitrates in food, 41
Nitrobenzol, 749, 750
Nitrogen compounds in milk, 111
Nitrogen, determination of, 61, 63
Nitrogen free extract, 48
Nitrogenous bodies
classification of, 36
separation of, in cheese, 160
in meat, 180
in milk, 111
Noodles, 266
Notification, 9
Noyau, 605
Nuclein, 40
Nutmeg, 360, 361
adulteration of, 362
Macassar, 363
microscopical structure of, 362
oil of, 747
standard, 362
Nutrose, 121
Nuts, composition of, 216

Oats, 213, 218, 242, 244
starch in, 221
Oil calorimeter, 387, 388
Oil, bitter almond, 747, 748
cassia, 327, 347
clove, 747
cocoanut, 429
corn, 422
cottonseed, 417
ginger, 346, 747
lard, 452
lemon, 742, 744, 745
lemongrass, 745
mace, 747
mustard, fixed, 354, 426
volatile, 353, 357
nutmeg, 747
oleo, 439
olive, 412
orange, 747
peanut, 423
peppermint, 747
poppyseed, 427
rape, 421
rosin, 428
sesame, 419
spearmint, 747
sunflower, 427
wintergreen, 747
Oils, edible, 368. See also Fats
acetyl value, 400
analysis of, 370
bromine absorption of, 385
bromination test, 386
cholesterol in, 404, 406, 408
constants of, 410, 411
elaidin test, 402
fatty acids in, 375, 377, 402, 403
iodine absorption of, 380, 383, 384
judgment as to purity of, 370
Maumené test, 386
melting point, 374
microscopical examination of, 409
phytosterol in, 404, 406, 408
rancidity of, 370, 431
references on, 457
refractometer for, 389
Reichert-Meissl number, 375
saponification of, 369, 379
sitosterol in, 423
specific gravity of, 371
factors, 372

Oils, edible, thermal tests, 385
titer test, 403
unsaponifiable matter in, 403
viscosity of, 374
Oils, essential, 745
Oleomargarine, 438
adulteration of, 451
constants of, 441
healthfulness of, 440
how distinguished from butter, 440, 442
manufacture of, 439
microscopical structure of, 449
odor and taste, 441
Zega's test for, 450
Oleo oil, 439
Olive, composition, 412
Olive oil, 412
adulteration of, 413, 416
examination of, 416
refraction of, 415
standard, 414
Olives, pickled, 711
Olive stones, 338
Orange colors, 630, 654
extract, 748
oil, 747
Orchil, 637, 642
O'Sullivan's sugar methods, 114, 489
Ovalbumin, 204
Oven, drying, 20
McGill's, 481
Ovomucin, 204
Ovomucoid, 205
Oxygen absorbed, 317
equivalent, 318
Oysters, 200

Palas rapeseed oil test, 422
Paprika, 341
Paraffin in confectionery, 519
in fats, 409
in honey, 516
in oleomargarine, 451
Pepper, long, 340
microscopical structure of, 335
nitrogen in, 334
in ether extract, 335
olive stones in, 338
piperin in, 331
determination of, 335
red. See Cayenne
shells, 337
standard, 337
white, 331
Peppermint oil, 747
Peptones, 38
in cheese, 161
in meat, 165, 181
in milk, 111
Perry, 553
Persian berries, 636
Petroleum ether, 58
Phloroglucide, 226
Phloroglucin, 225
Phosphate baking powders, 255
Phosphoric acid in baking chemicals, 265
in beer, 588
Phosphotungstic acid method for nitrogen separation, 182
reaction, 37
Photomicrography, 81
camera for, 84
Phytalbumose, 38
Phytolacca, 636
Phytosterol, 404, 408
Piccalilli, 711
Pickled meats, 172
Pickles, 708, 711
adulteration of, 711
Pickling pump, 173
Pineapple essence, 752, 753
Pioscope, 128
Piotrowski's reaction, 37
Piperin, 331
determination of, 335
Plant crystals, 78
Plasmon, 122



Polarization of wine, 570
Poppyseed, 427
oil, 427
Pork, composition of, 170
cuts of, 170
Porter, 576
Port wine, 557
Potash determination, 264
Potassium myronate, 353, 357
Potatoes, 214
proteids of, 233
starch of, 222
Poultry, composition of, 170
Preparation of sample, 49
Preservatives, 661
commercial food, 663
in butter, 436
in canned goods, 705, 706
in fish, 201
in meats, 174, 184
in milk, 137, 143
in table sauces, 710
of eggs, 208
references on, 679
regulation of, 662
Pressure pump, 18
Process butter, 442
Proof spirit, 547
Prosecution, 9
Proteids, 36
insoluble, 39
of barley, 218, 233
of beer, 588
of cereals, 228
of condensed milk, 150
of eggs, 205
of milk, 80
calculation of, 117
determination of, 109
of peas, 233
of potatoes, 233
of rye, 218, 232
of wheat, 218, 230
Protein, 48
factor for, 48
Protein grains, 78
Proteolytic fermentation, 157
Proteoses, 38, 220
Proto-albumose, 38
Proximate analysis, extent of, 47
expression of results of, 48
Prussian blue, 638
Ptomaines, 172
Publication of adulterated foods, 9
Pulfrich refractometer, 389
Pycnometer, 51
Pyroligneous acid, 625

Quassiin, 590
Quercitannic acid, 318
Quevenne's lactometer, 95
Quince essence, artificial, 752, 753
Quotient of purity of sugar, 481

Raffinose, 219, 472
determination of, 503
Rancidity, 370, 431
Rape oil, 421
test for, 422
seed, 421
Raphides, 78
Reagents, 31
references on, 34
table of, 24–30
Red colors, 631, 636, 652
Red ocher in sausages, 190
Red pepper. See Cayenne
Red wines, 554
Redwood, 345
References on beer, 607
butter, 458
cereals, 273
cocoa, 308
coffee, 308
colors, 660
dietetics, 440
distilled liquors, 608
eggs, 211
flavoring extracts, 754
food economy, 44
inspection, 10
fruits, 273
immersion refractometer, 767
laboratory equipment, 34
leavening materials, 275
microscope, 86
milk, 163
oils, 457
reagents, 34
spices, 366
sugars, 522
tea, 308
vinegar, 626
wine, 608
Refractometer, 389
Abbé, 389, 397
Amagat and Jean, 389
butyro, 300, 443
critical line, 443
heater for, 301
immersion, 757, 758
Pulfrich, 389
sliding scale for, 306
tables for, 105, 394, 398, 759, 763, 764
Wollny, 103, 389
Reichert-Meissl method, 375
Reichert number of butter, 445
Reinsch's test for arsenic, 592
Relishes, 708
Renard's test for peanut oil, 424
for rosin oil, 428
Renovated butter, 438
Resins, 77
Respiration calorimeter, 2

Rice, 213
starch, 222
Richmond's cane sugar method, 145
sliding milk scale, 117
Ritthausen's method for milk proteids, 109
Roeser's mustard oil method, 357
Ropy milk, 94
Roquefort cheese, 158
Rosin oil, 428
Rota's color scheme, 644
Rubner's fuel value factors, 43
Rum, 599
essence, 600
standards, 599
Rye, 213, 242
proteids of, 218, 232
starch, 221

Saccharimeter, 473
double wedge, 476
forms of, 478
normal weights for, 478
scales compared, 478
single wedge, 474
Soleil-Ventzke, 473
triple shadow, 476
Saccharimetry, 473
Saccharin, 682
detection of, 683
determination of, 684
Saccharine products, 461
Saccharoses, 461
Safflower, 637
Saffron, 637
Sago, 223
Saleratus, 258
Salicylic acid, 671
detection of, 143, 671
determination of, 672
in meat, 174, 185
in milk, 143
Salted meats, 172
Sanatogen, 122
Sanose, 122
[redacted]

Sesame oil, adulteration of, 420
tests for, 420
seeds, 419
Sherry wine, 557
Shredded wheat, 266
Sieve tissue, 77
Silent spirit, 593
Sinalbin, 353
mustard oil, 353
Sinigrin, 354
Sinks, 15
Sitosterol, 423
Smoked meats, 172
"Soaked" goods, 707
Soda, determination of, 264
Sodium benzoate, 673
bicarbonate, 258
bisulphite, 675
carbonate, in milk, 142, 144
hydroxide, tenth normal solution, 31
salicylate, 671
Soleil-Ventzke saccharimeter, 473
Sorghum, 468
Souring of milk, 93
Sour milk, 103
Soxhlet extractor, 57
Soxhlet's milk sugar method, 114, 116
Spaghetti, 260
Sparkling wine, 555
Spearmint oil, 747
Specific gravity bottle, 51
of beeswax, 515
of liquids, 49
of liquors, 527
of milk, 95, 115
serum, 129
temperature correction for, 97
of oils, 371
of vinegar, 614
Specific rotary power, 479
Spent tea leaves, 285
Spices, 310
adulterants of, 315
[redacted]

Standards for butter, 433
cassia, 330
cayenne, 344
cheese, 158
cinnamon, 330
cloves, 320
cocoa, 304
cream, 155
ginger, 350
grape sugar, 469
lard, 453
lemon extract, 739
mace, 364
meats, 172
milk, 124, 126
molasses, 507
mustard, 359
nutmeg, 362
olive oil, 514
pepper, 337
renovated butter, 438
rum, 509
sugars, 462, 469
vinegar, 619
wine, 559
whiskey, 596
Standard solutions, equivalents of, 32
refractometric readings of, 764
Starch, 77, 219
arrowroot, 222
barley, 221
bean, 222
buckwheat, 222
classification of, 220
corn, 221
detection of, 219
determination of, 223
by acid conversion, 223
by diastase method, 223
in baking powder, 262
in cereals, 228
in milk, 146
in sausages, 185
in spices, 313
oat, 221
pea, 222
potato, 222
rice, 222
rye, 221
sago, 223
syrup, 470
tapioca, 222
under polarized light, 223
wheat, 221
State control, 1, 3, 5, 9
references on, 10
Stearin, beef, 455
cottonseed, 418
lard, 453
Sterilized butter, 438
Still, alcohol, 530
fractionating, 59
Still, nitrogen, 63
water, 20
Still wine, 555
Stilton cheese, 158
Stokes' milk centrifuge, 100
Stone's method of carbohydrate separation, 227, 228
Storch's proteid, 91
Strippings, 92
Stutzer's gelatin method, 183
Suberin, 77
Sucrate of lime, 155
Sucrose. See Cane sugar
Suction pump, 17
Suet, 430
Sugar, 461
analysis of, 480
beet, 465
cane, 462, 463
classification of, 461
grape. See Dextrose
in fruits, 462
maple, 466, 767, 769
muscovado, 464
organic non sugars in, 481
quotient of purity, 481
raw, 463, 464
references on, 522
refining, 466
standards, 462, 469
ultramarine in, 466, 498
Sulphuric acid
in baking chemicals, 265
in vinegar, 617
Sulphuring, 675
Sulphurous acid, 675
detection of, 675
determination of, 676
in meat, 174, 184
Sunflower oil, 427
seeds, 428
Sweeteners, artificial, 682
Sweet wine, 555
Syrup, analysis of, 498
maple, 466, 769
mixing, 470
starch, 470

Table sauces, 709
preservatives in, 710
Tallow, 430
Tannin in cloves, 317
in tea, 281
in wine, 571
Tapioca, 222, 223
Tartaric acid in baking powder, 261
in fruit products, 722
Tartrate baking powders, 257
Tea, 276
adulteration of, 284
analytical methods, 279
ash of, 278, 280
astringents in, 287

Tea, caffeine in, 280
exhausted leaves in, 285
extract of, 283
facing of, 284
foreign leaves in, 285
leaf, characteristics of, 286
microscopical examination of, 288
references on, 308
spent leaves in, 285
stems in, 286
tablets, 287
tannin in, 281
theine in, 280
Teller's method of separating wheat proteids, 231
Theine, 280
Theobromine, 302, 303
Tin, action of fruits and vegetables on, 695, 607
determination of, 703, 704
salts in molasses, 507
Titer test, 403
Tocher's sesame oil test, 420
Tomato ketchup, 708
coloring in, 709
preservatives in, 708, 710
Tonka bean, 732
tincture, 732
Turmeric, 350
as an adulterant, 352
microscopical structure of, 351
tests for, 353, 636, 637

Ultramarine blue, 638
in sugar, 466, 498
Uno beer, 581

Vacuoles in yeast cells, 252
Vanilla bean, 729, 730
exhausted, 731
Vanilla extract, 729
adulteration of, 733
alcohol in, 732, 738
alkali in, 732
analysis of, 731
Vaporimeter, 545
Veal, composition of, 168
cuts of, 168
Vegetable colors, 635, 642
in sausages, 190
Vegetables, 216
ash of, 233
composition of, 214
methods of approximate analysis of, 217
references on, 273
Ventilation, 13
Villivecchia and Fabris' sesame oil test, 420
Vinegar, 609
acidity of, 615
acids of, 616
adulterated, 624
adulteration of, 619, 620
alcohol in, 616
analysis of, 614
arsenic in, 626
artificial, 620
ash of, 621
solubility and alkalinity of, 614
beer, 612
caramel in, 625
cider, 610
artificial, 620
composition of, 610
copper in, 626
distilled, 613
extract of, 614
furfurol in, 625
glucose, 613
hydrochloric acid in, 617
lead in, 625
lead acetate test for, 617, 625
levulose in, 619
malic acid in, 617
malt, 612
manufacture of, 610
metallic impurities in, 625
mineral acids in, 616, 617
molasses, 613
nitrogen in, 615

Vinous fermentation, 524
Viscogen, 156
Viscosity of cream, 156
of oils, 374

Walnut ketchup, 709
Water-bath, 19
Water glass, 208
Waterhouse butter test, 446
Weiss beer, 576
Werner-Schmidt method for fat in cheese, 160
in milk, 103
Westphal balance, 50
Wheat, 213
ash of, 234
proteids of, 218, 230
shredded, 266
starch, 221
Whiskey, 594
adulteration of, 596
artificial, 596
Bourbon, 595
composition of, 595
Irish, 595, 597
manufacture of, 594
standards, 596
Wijs's iodine absorption method, 384
Wild's saccharimeter, 478
Wiley's bromine pipette, 387
method for nitrogen separation in meat, 182
Wiley and Ewell's double dilution sugar method, 113, 503
Wine, 554
acidity of, 564
added alcohol in, 563
adulteration of, 560
analytical methods for, 564
Burgundy, artificial, 560
California, 558
cane sugar in, 561
Cazeneuve's color method, 572
chaptalizing, 561
claret, 557
artificial, 560
classification of, 555
coloring matter in, 571, 573
composition of 556
cream of tartar in, 570
"dry," 555
Dupré's color method, 572
extract in, 564, 565 567
fortified, 555
fruit other than grape, 563
glycerin in, 570
hock, 559
Madeira, 557
Malaga, artificial, 560
Wine, malic acid in, 569
manufacture of, 554
natural, 555
non-volatile acids in, 568
plastering, 560
polarization of, 570
port, 559
potassium sulphate in, 571
red, 554
reducing sugar in, 570
references on, 608
sherry, 557
artificial, 560
sparkling, 555
standards, 559
still, 555
sweet, 555
tannin in, 571
tartaric acid in, 568
varieties of, 557
vinegar, 611
volatile acids in, 564
watering of, 562
white, 554
yeast of, 554
Wintergreen, oil of, 747
Wollny milk fat refractometer, 103
tables for using, 105
table for converting Wollny degrees into n_D, 107
Wood vinegar, 614, 625
Wool, double dyeing method with, 641
dyeing of, 640
for color tests, 640
vegetable colors on, 642

Xanthin, 165
Xantho-proteic reaction, 36
Xylan, 226
Xylose, 225

Yeast, 249, 251
adulteration of, 253
ash of, 250
compressed, 250
dry, 250
in cider, 548
in wine, 554
microscopical examination of, 250
starch in, 253
testing, 250
vacuoles in, 252
Yellow colors, 631, 636, 654

Zega's test for oleomargarine, 450
Zinc salts, 699
determination of, 703

CEREALS.

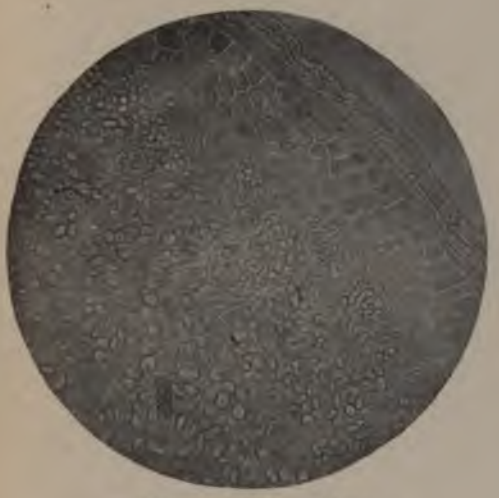


FIG. 121.—Barley, ×110.
Transverse section, showing in order, pericarp, seed coats, aleurone layer, and starch cells.



FIG. 122.—Barley, ×55.
Surface view of epidermis with hairs.

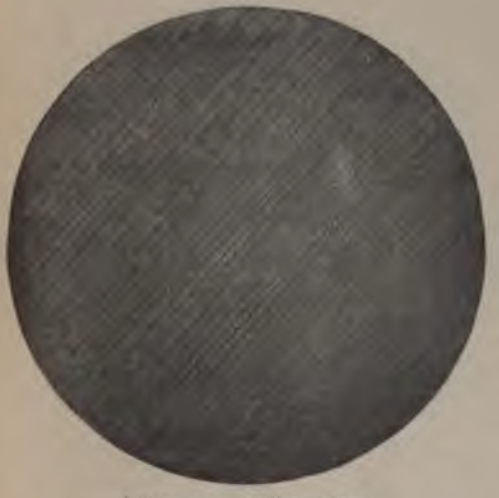


FIG. 123.—Barley, ×125.
Surface view of upper chaff layer.

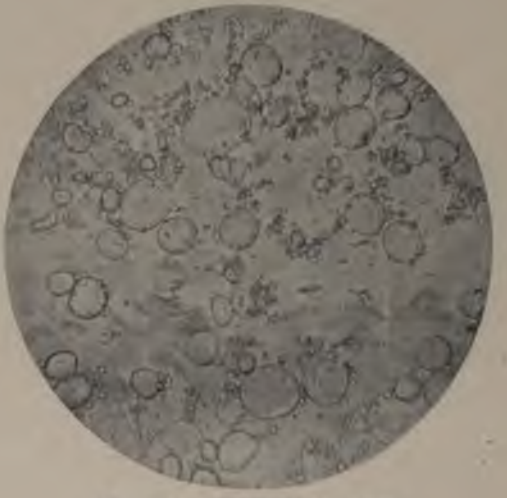


FIG. 124.—Barley Starch, ×220.



CEREALS.

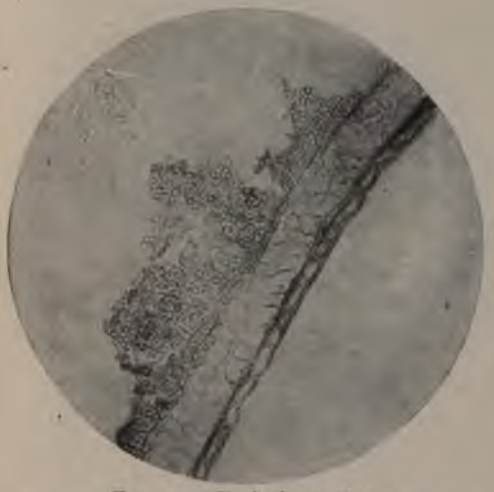


FIG. 125.—Buckwheat, ×110.
Transverse section through part of pericarp, seed coat, and part of endosperm.

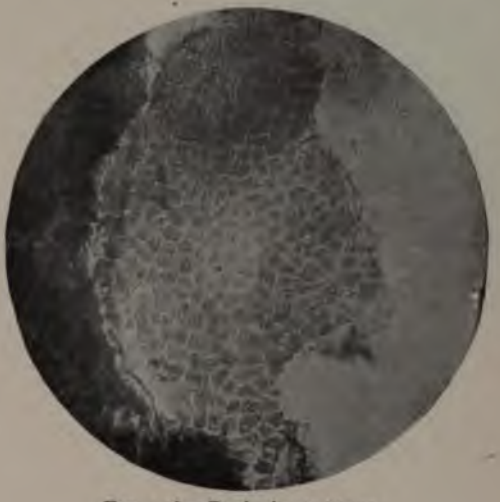


FIG. 126.—Buckwheat, ×110.
Surface view of scutellum.

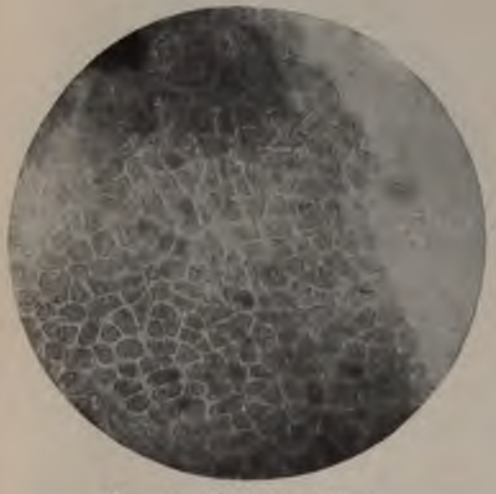


FIG. 127.—Buckwheat, ×110.
Surface section. Aleurone or proteid layer.

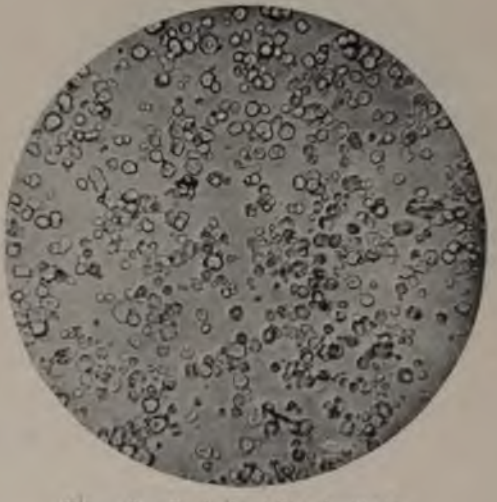


FIG. 128.—Buckwheat Starch, ×220.
Starch granules separated.

CEREALS.



FIG. 129.—Buckwheat Starch, ×110.
Starch grains in masses.



FIG. 130.—Corn, ×110.
Transverse section through pericarp, seed coat, proteid layer, and part of endosperm, showing starch cells.



FIG. 131.—Corn, ×110.
Surface view showing two layers of the mesocarp.

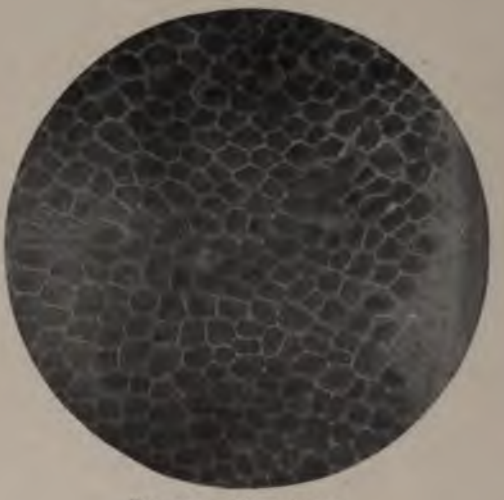


FIG. 132.—Corn, ×110.
Surface section. Proteid layer.

CEREALS.

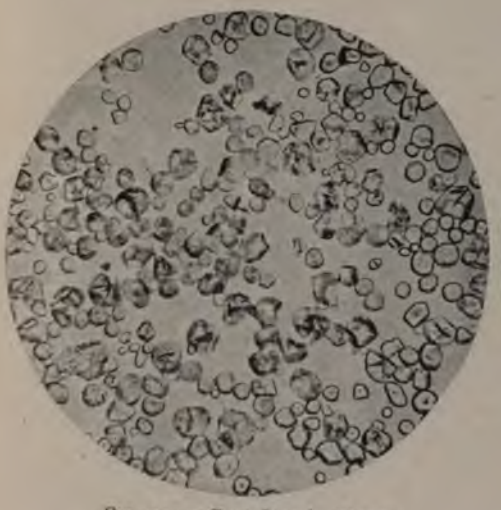


FIG. 133.—Corn Starch, ×220.

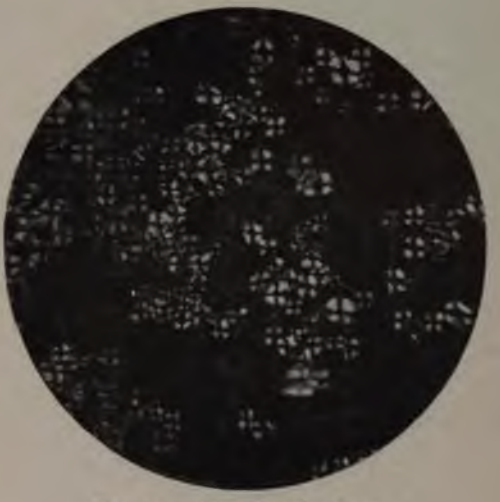


FIG. 134.—Corn Starch, ×220.
With polarized light.

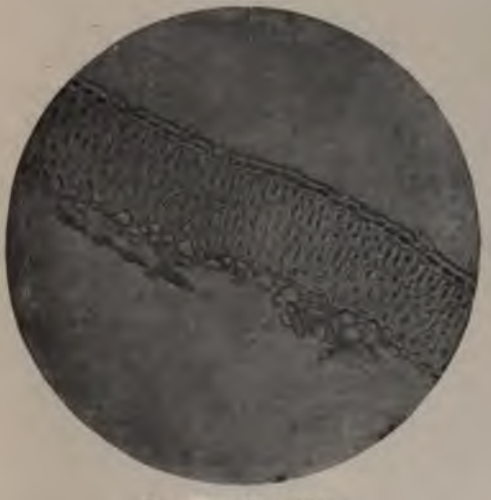


FIG. 135.—Oat, ×110.
Transverse section through chaff.



FIG. 136.—Oat, ×110.
Surface section. Proteid layer with fragments of epidermis and hairs.

CEREALS.

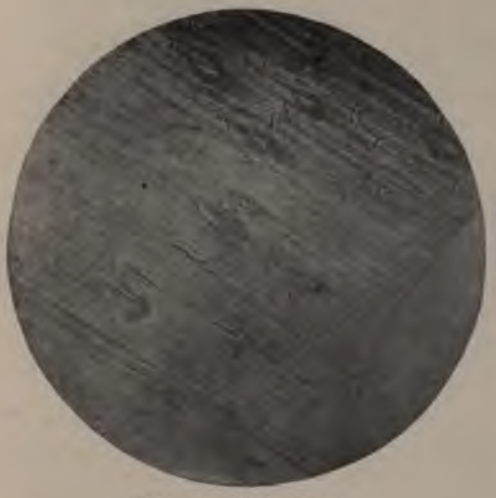


FIG. 137.—Oat, ×110.
Surface view of upper chaff layer.



FIG. 138.—Oat ×55.
Surface view of epidermis and hairs.

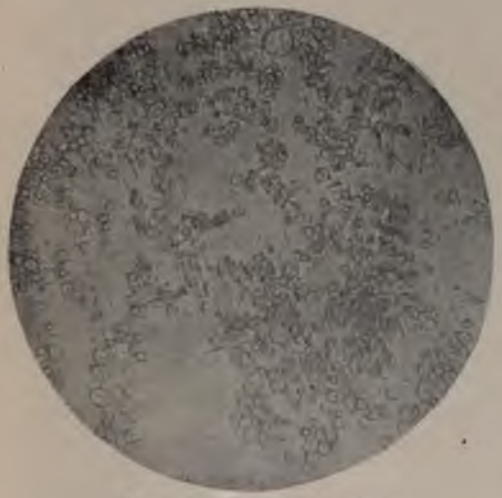


FIG. 139.—Oat Starch, ×220.

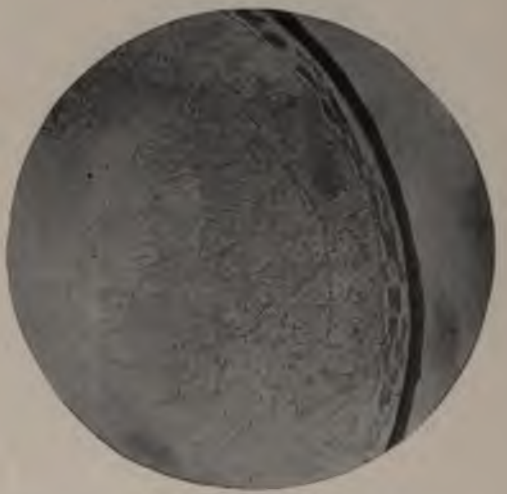


FIG. 140.—RICE, ×110
Transverse section through seed coat and part of endosperm

CEREALS.



FIG. 141.—Rice, ×110.
Surface section through starch cells.

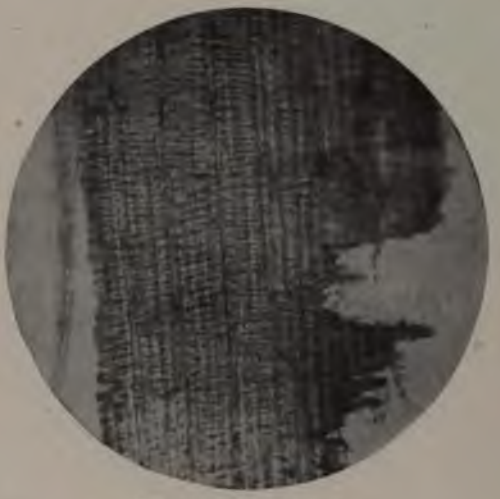


FIG. 142.—Rice, ×110.
Surface view of upper chaff layer

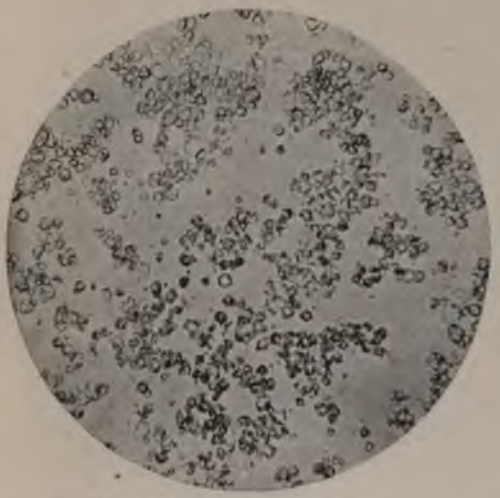


FIG. 143.—Rice Starch, ×220.



FIG. 144.—Rye, ×18
Transverse section through the entire grain.

PLATE VII.

CEREALS.



FIG. 145.—Rye, ×110.
Transverse section through pericarp, seed coat, aleurone layer, and starch cells of endosperm.

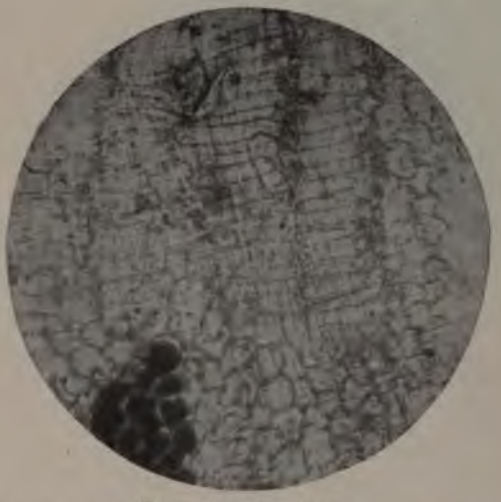


FIG. 146.—Rye, ×110.
Surface view of epidermis and underlying layers.

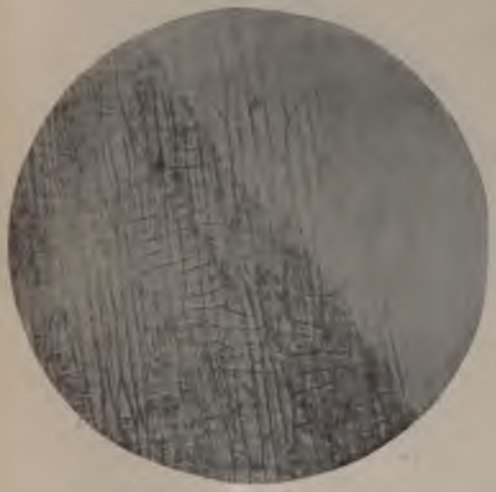


FIG. 147.—Rye, ×110.
Surface view of epidermis and of seed coat.

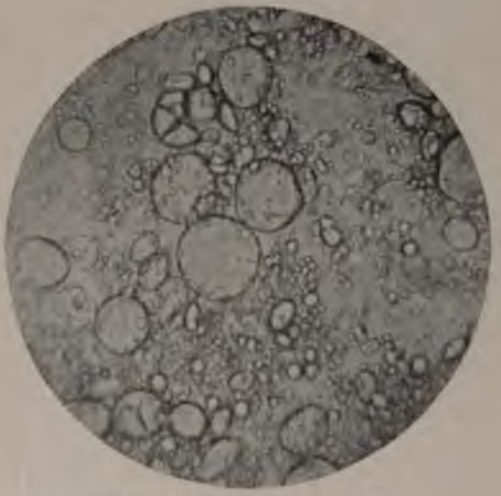


FIG. 148.—Rye Starch, ×220.

CEREALS.

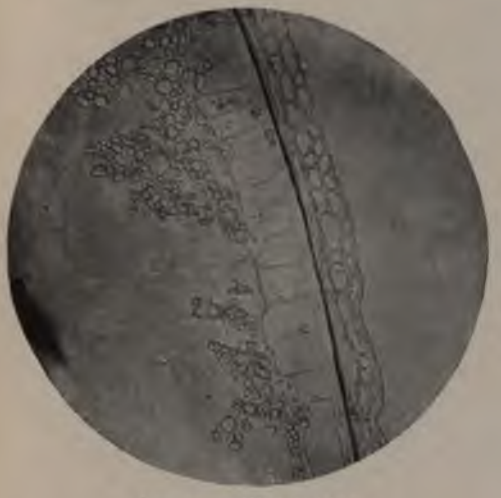


FIG. 149.—Wheat, ×110.
Transverse section through pericarp, seed coat, proteid layer, and starch cells of endosperm.



FIG. 150.—Wheat, ×110.
Surface view of outer and inner epidermis showing proteid layer. Also



FIG. 151.—Wheat, ×110.
Surface view of epidermis, with hairs.

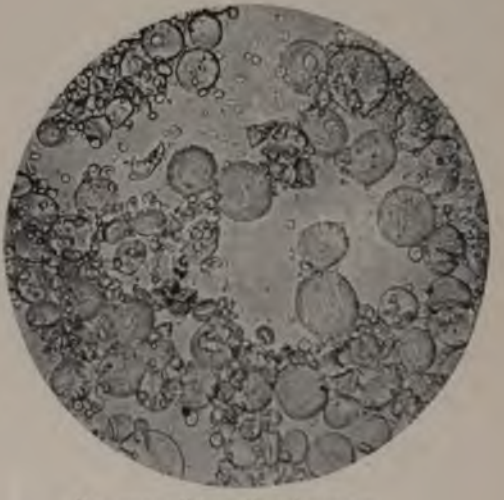


FIG. 152.—Wheat Starch. ×220.

LEGUMES.

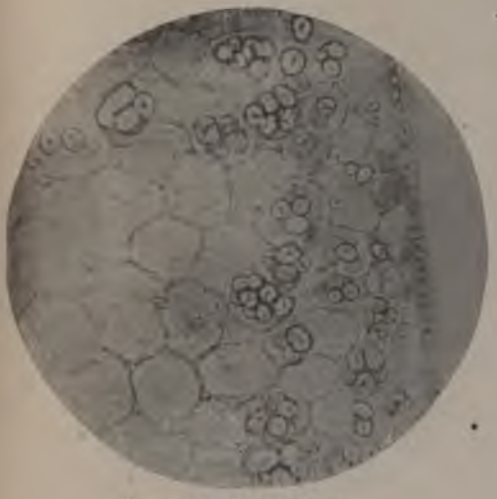


FIG. 153.—Bean, ×110.
Transverse section through starch cells.

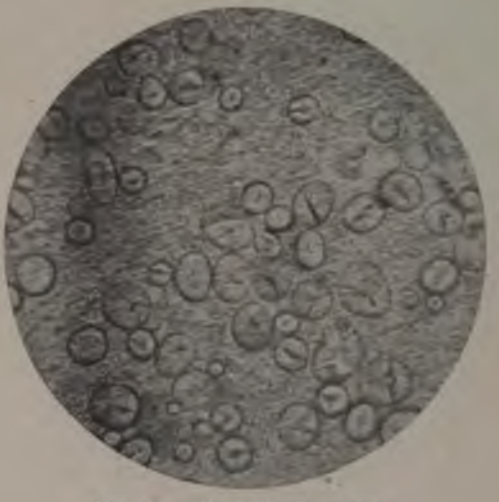


FIG. 154.—Bean Starch, ×220

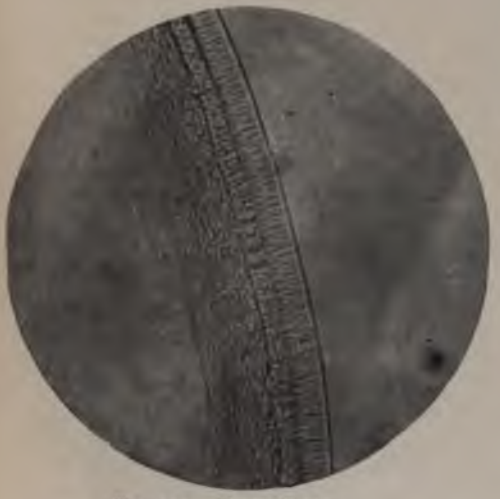


FIG. 155.—Bean, ×110.
Transverse section through hull, showing palisade cells of epidermis, and underlying hypoderma.

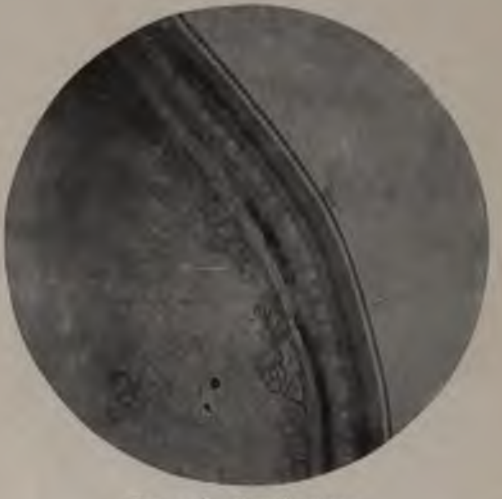


FIG. 156.—Lentil, ×110.
Transverse section through hull and part of endosperm, showing some of the starch cells.

LEGUMES.



FIG. 157.—Lentil, ×110.
Surface view of epidermis.



FIG. 158.—Pea, ×110.
Transverse section through hull and seed coat, showing outer palisade cells and underlying hypoderma.



FIG. 159.—Pea. ×110.
Surface section through base of palisade layer.

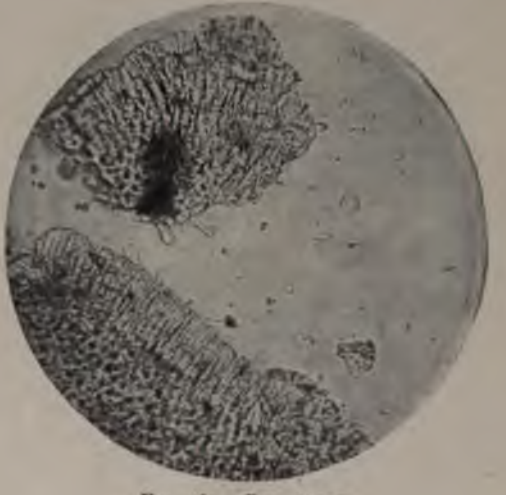


FIG. 160.—Pea, ×110.
Powdered pea hulls.

LEGUMES.



FIG. 161.—Pea, ×110.
Surface view of palisade cells.



FIG. 162.—Pea, ×110.
Transverse section through starch cells.

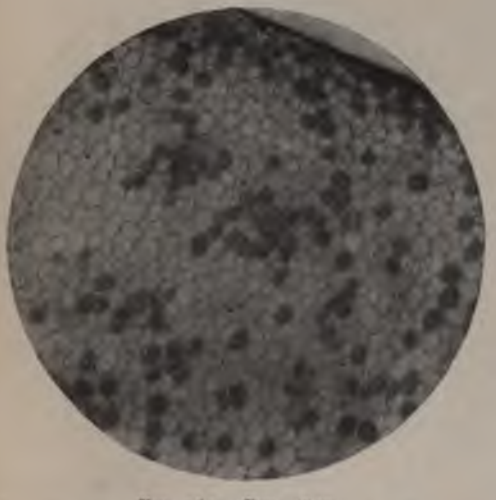


FIG. 163.—Pea, ×30.
Transverse section through starch cells.

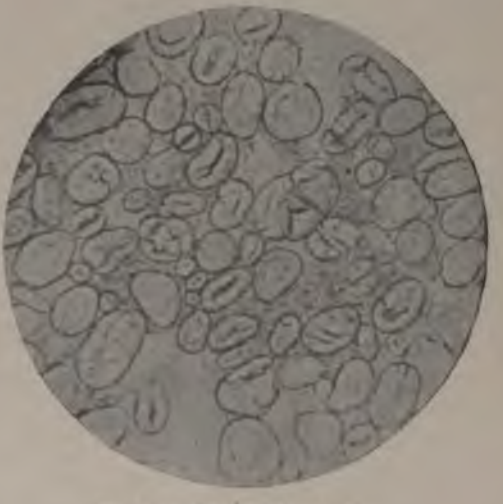


FIG. 164.—Pea Starch ×220.

PLATE XII.

MISCELLANEOUS STARCHES.

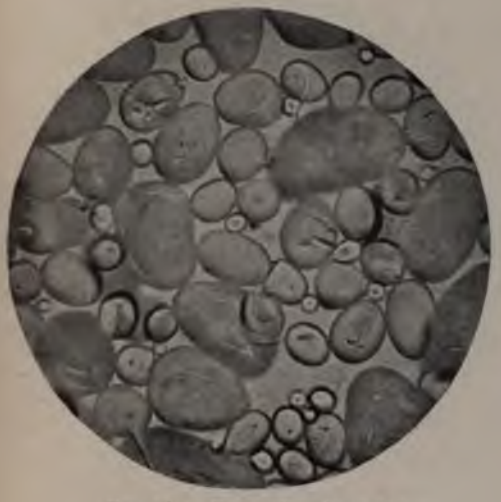


FIG. 165.—Potato Starch, ×220.

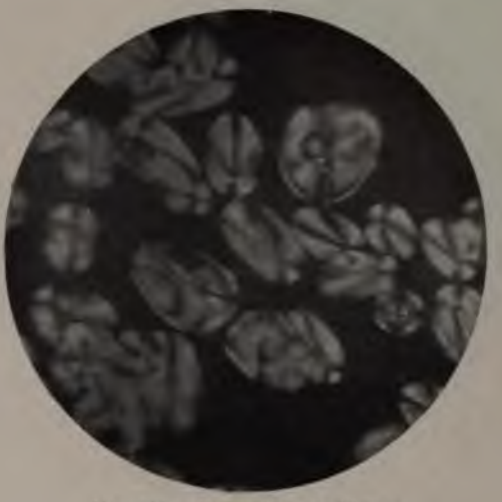


FIG. 166.—Potato Starch, ×220.
With polarized light.

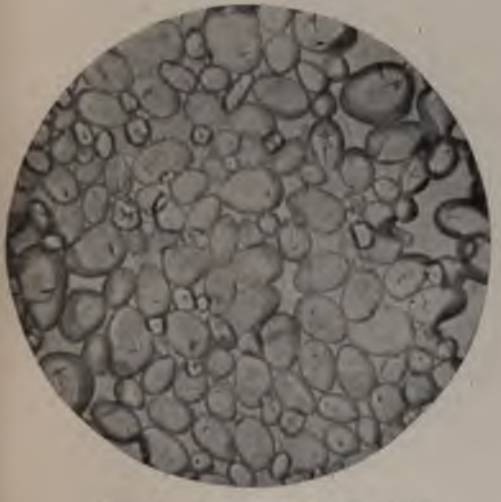


FIG. 167.—Arrowroot Starch, ×220.

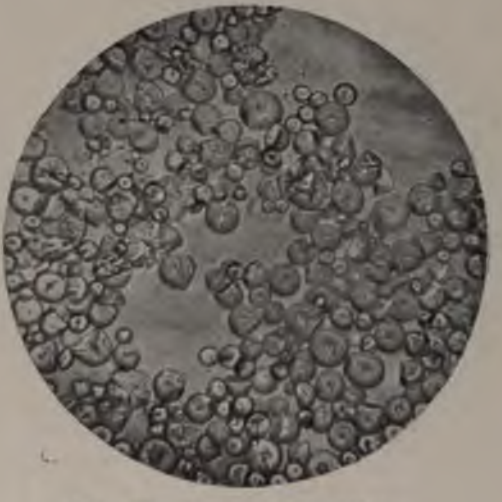


FIG. 168.—Tapioca Starch, ×220.
(Cassava.)

TURMERIC. SAGO.

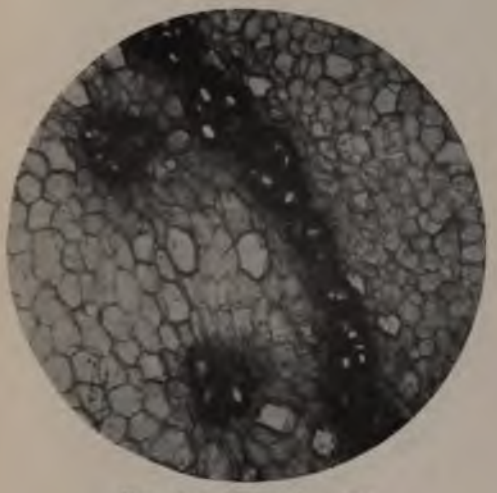


FIG. 169.—Turmeric, ×70.
Transverse section through rhizome.

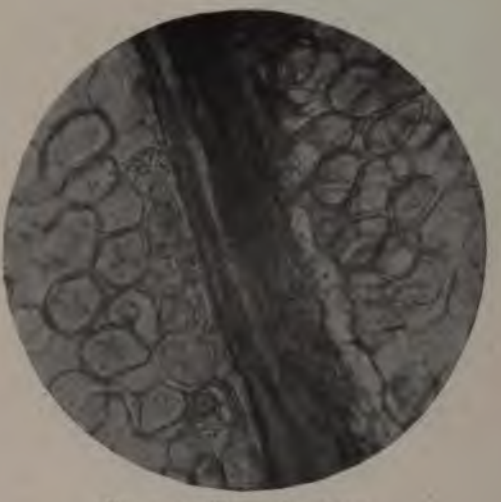


FIG. 170.—Turmeric, ×110.
Longitudinal section. Note spiral ducts through the center.

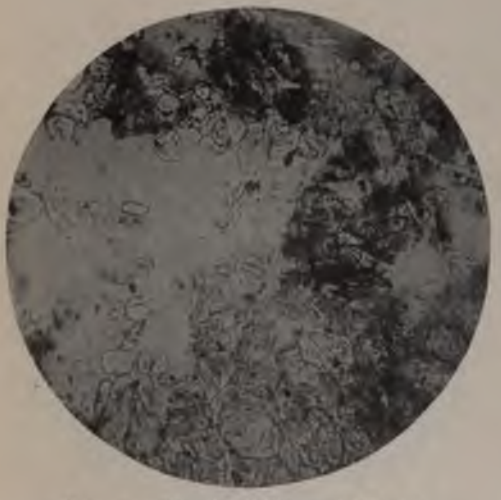


FIG. 171.—Powdered Turmeric, ×110.
Showing starch grains, fragments of cell tissue, coloring matter, etc.

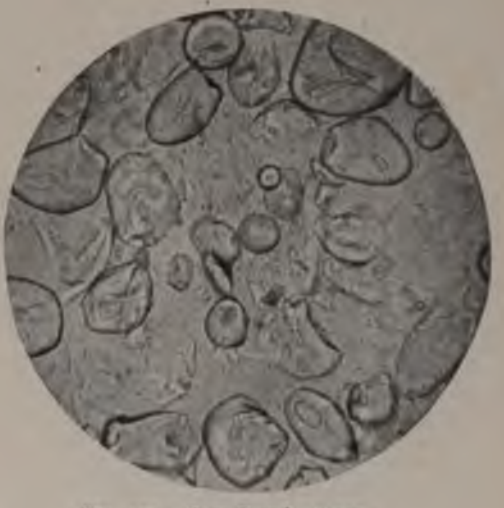


FIG. 172.—Sago Starch, ×220.

COFFEE.

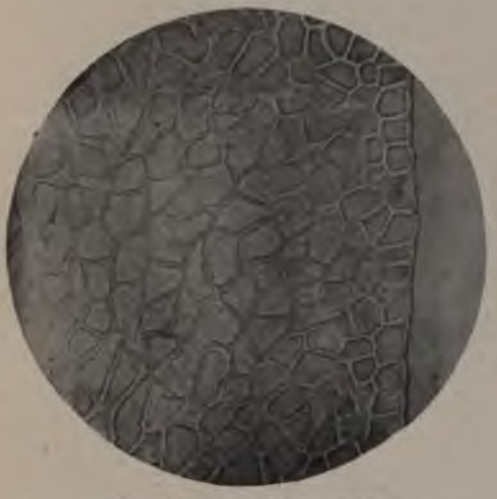


FIG. 173.—Raw Coffee, ×110.
Transverse section of outer portion of endosperm.

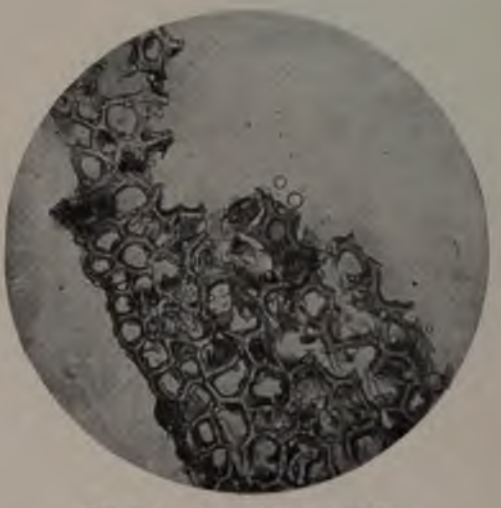


FIG. 174.—Roasted Coffee, ×130.
Transverse section through parenchyma of endosperm.

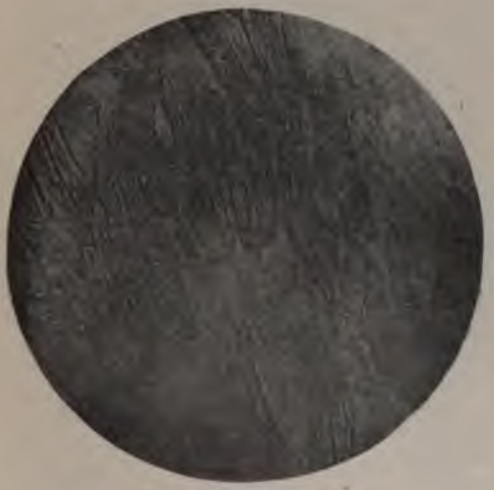


FIG. 175.—Coffee, ×110.
Surface view of seed coat.

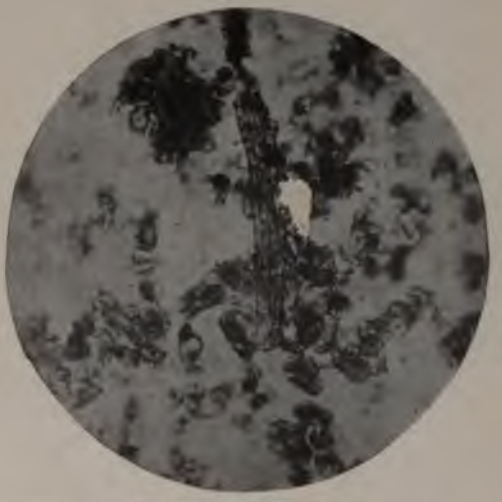


FIG. 176.—Coffee, ×110.
Roasted, ground coffee, showing fragments of endosperm parenchyma and of seed coat.

COFFEE. CHICORY.

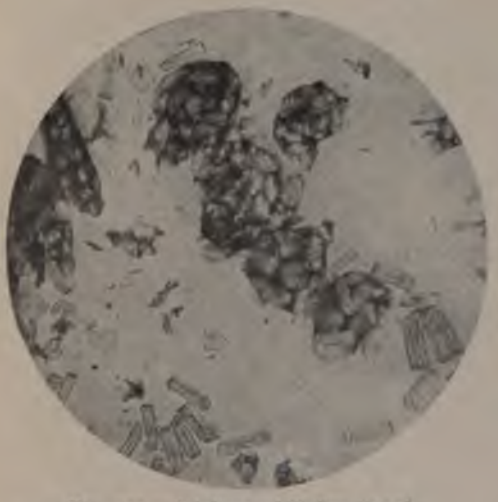


FIG. 177.—Adulterated Coffee, ×130.
Dark masses of roasted pea starch are shown, with transparent fragments of the palisade cells of the pea-hull.

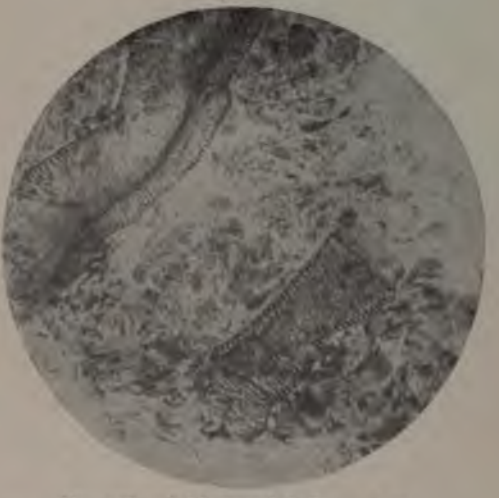


FIG. 178.—Adulterated Coffee, ×130.
The vascular ducts of chicory show most conspicuously in this field.



FIG. 179.—Chicory, ×25.
Transverse section through the root.

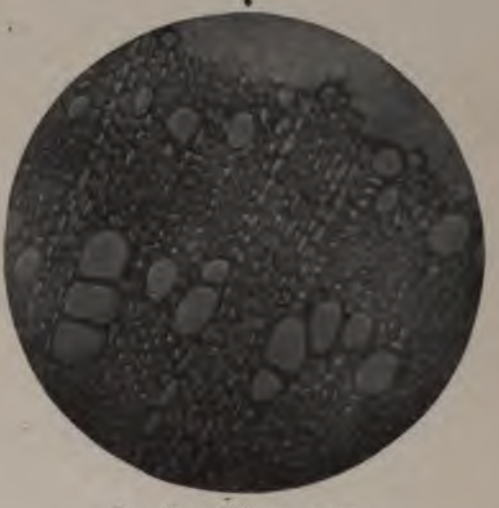


FIG. 180.—Chicory, ×110.
Transverse section.

CHICORY. COCOA.

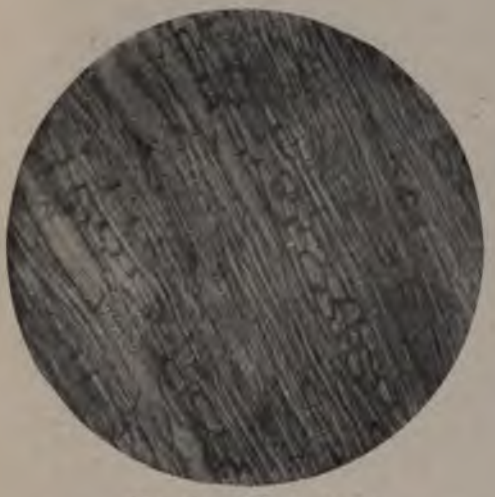


FIG. 181.—Chicory, ×110.
Tangential section, showing reticulated ducts and wood parenchyma.



FIG. 182.—Chicory, ×110
Radial section, showing bark parenchyma and milk ducts.

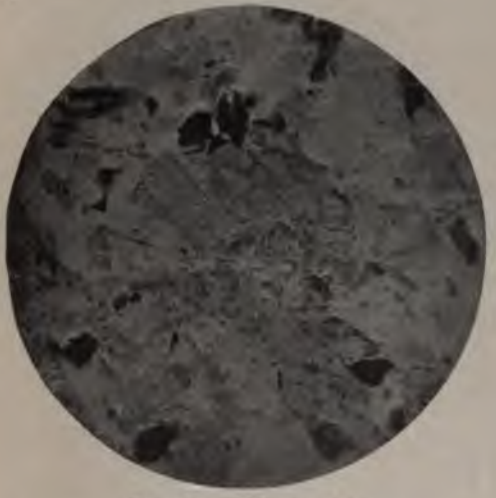


FIG. 183.—Chicory, ×110.
Roasted and ground, showing fragments of ducts and other tissues.



FIG. 184.—Cocoa, ×110.
Transverse section through periphery of seed, seed coats, and cotyledon.

COCOA.

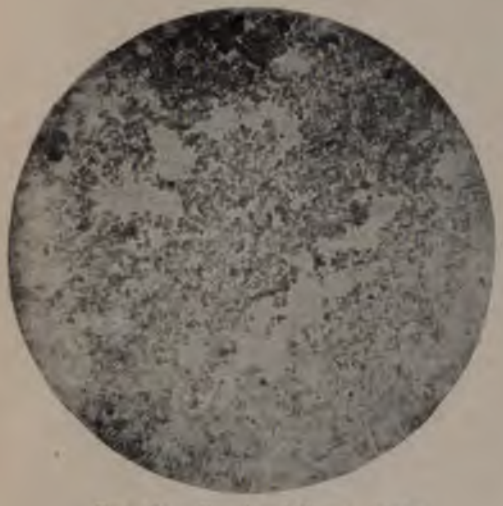


FIG. 185.—Powdered Cocoa, ×110.

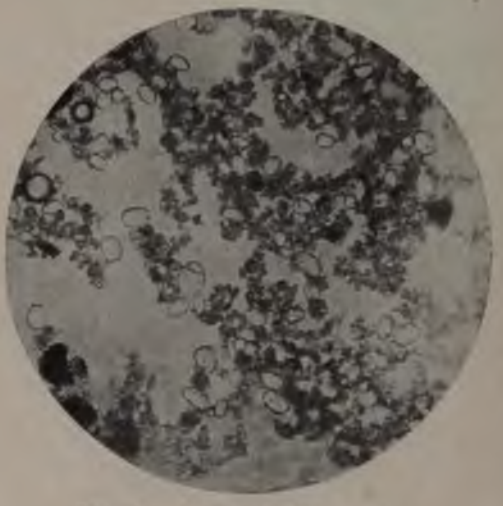


FIG. 186.—Adulterated Cocoa, ×110.
Showing admixture of arrowroot with the cocoa powder.

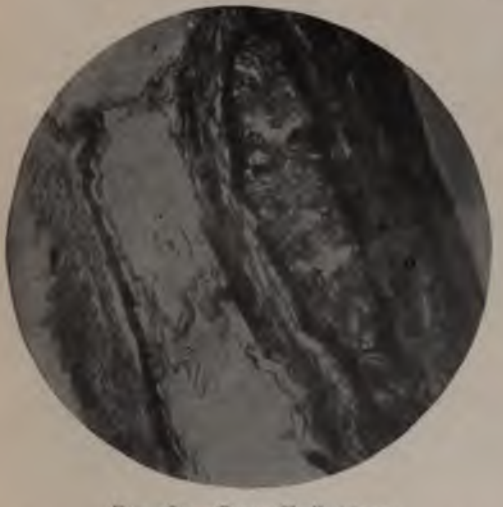


FIG 187.—Cocoa Shell, ×110.
Transverse section through epidermis, pulp, and mucilaginous layers of the pericarp and seed coat.



FIG. 188.—Cocoa Shell, ×110.
Longitudinal section through shell

TEA. SPICES.

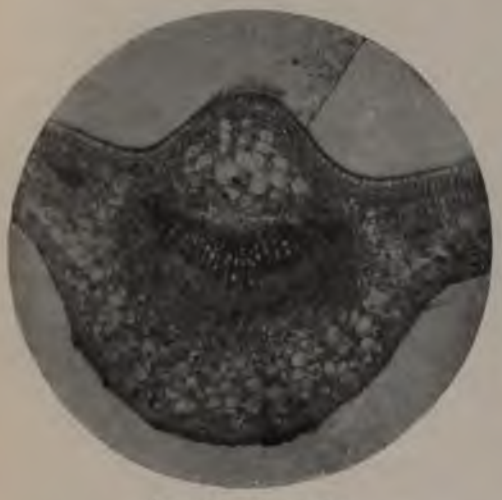


FIG. 189.—Tea, ×55.
Transverse section through midrib of leaf. Note the palisade layer below the upper epidermis, the inner wood vessels above the center, and the parenchyma of the pulp.

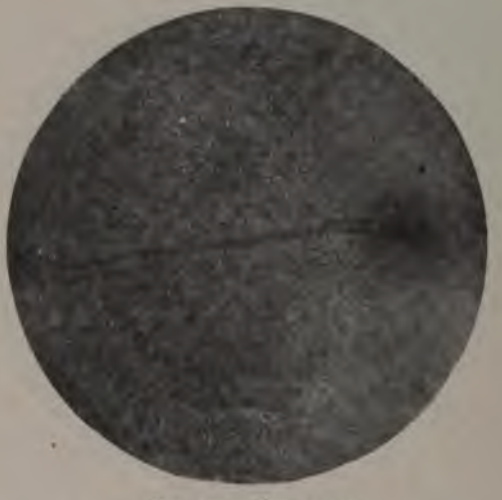


FIG. 190.—Tea, ×110.
Surface view of lower epidermis, with stomata and one of the hairs.



FIG. 191.—Allspice, ×9.
Transverse section through the entire berry, showing the two cells, with kidney shaped seed in each.

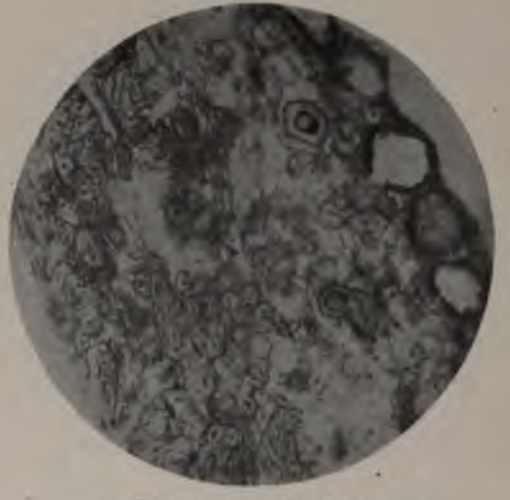


FIG. 192.—Allspice, ×70.
Transverse section through pericarp, showing oil spaces and stone cells.

SPICES.



FIG. 193.—Allspice Seed, ×110.
Transverse section through seed shell and part of embryo, showing starch cells.

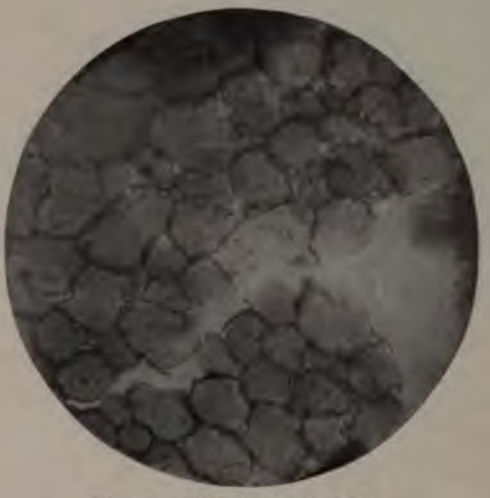


FIG. 194.—Allspice Seed, ×110.
Transverse section through the resinous portion of the seed coat, showing port wine colored lumps of gum or resin.

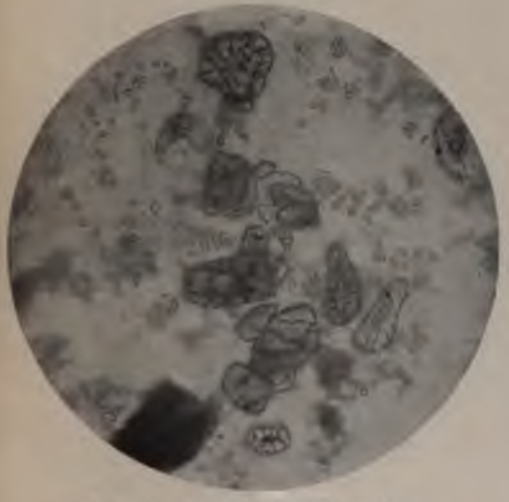


FIG. 195.—Powdered Allspice, ×110.
Showing stone cells, resinous lumps, and starch.

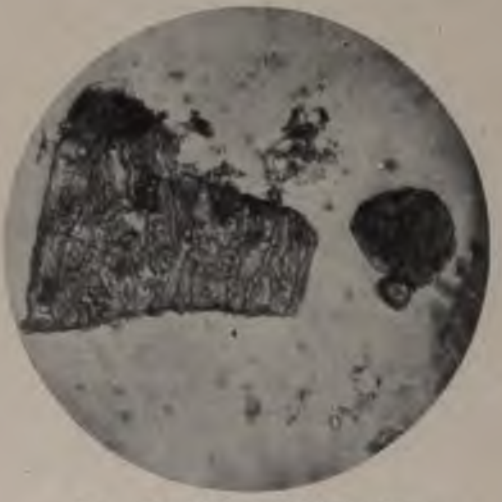


FIG. 196.—Adulterated Allspice, ×110.
Showing a large fragment of the seed skin of cayenne at the left.

SPICES.

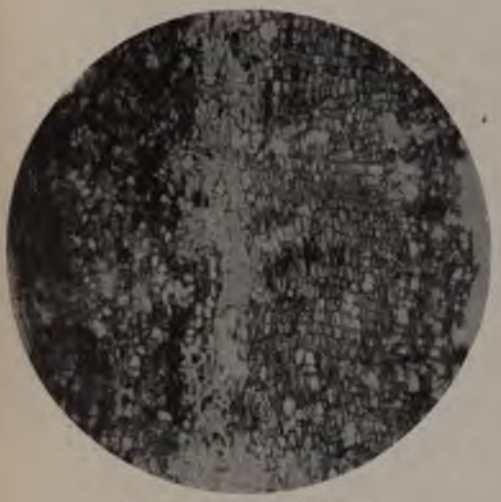


FIG. 197.—Cassia Bark, ×45.
Transverse section through the bark.



FIG. 198.—Cassia Bark, ×45.
Longitudinal section.



FIG. 199.—Cassia Bark, ×110.
Transverse section, showing cork cells, parenchyma, and stone cells.

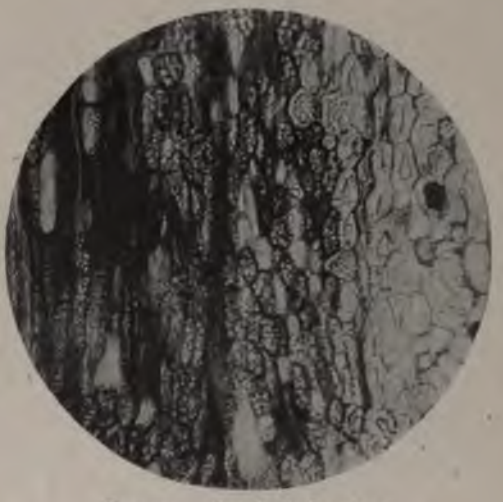


FIG. 200.—Cassia Bark, ×110.
Longitudinal section, showing bunches of bast fibers at the left, starch cells in the center, and stone cells at the right.

SPICES.

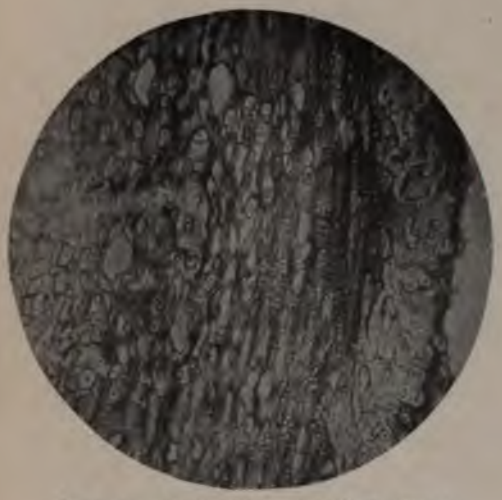


FIG. 201.—Ceylon Cinnamon Bark, ×110.
Transverse section, showing many bast fibers and starch cells.



FIG. 202.—Ceylon Cinnamon Bark, ×110.
Longitudinal section, showing bast fibers, stone cells, and parenchyma.

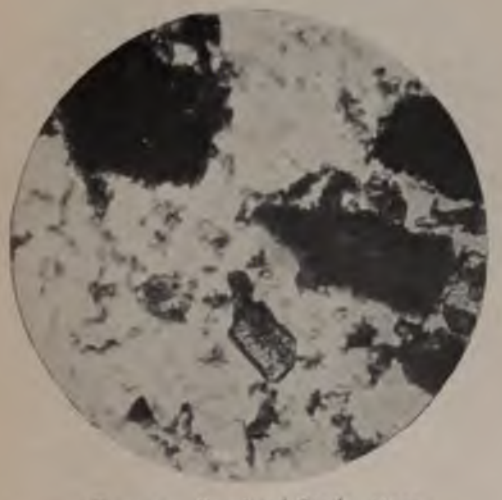


FIG. 203.—Powdered Cassia, ×110.
Showing stone cells, starch, and corky tissue.

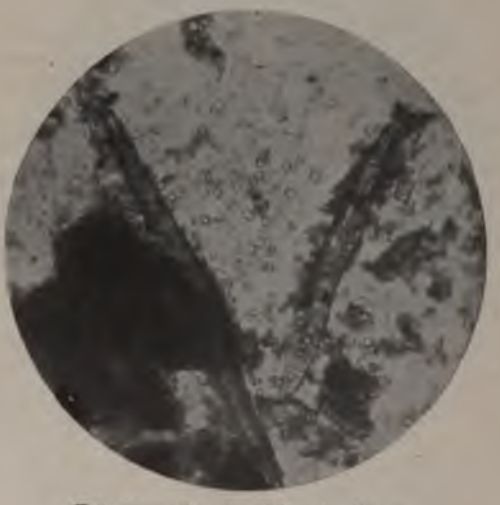


FIG. 204.—Powdered Cassia, ×110.
Showing bast fibers and starch.

SPICES.

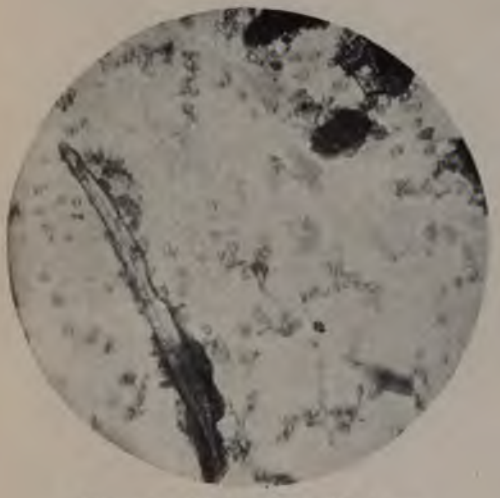


FIG. 205.—Powdered Cassia, × 110.
Showing large bast fiber and starch grains.

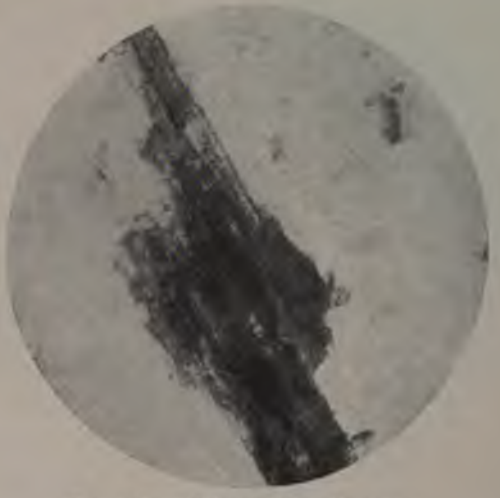


FIG. 206.—Adulterated Cassia, ×110.
A mass of foreign bark.

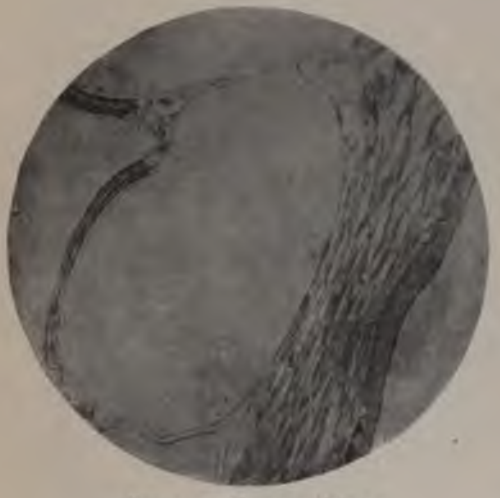


FIG. 207.—Cayenne, ×110.
Transverse section through pericarp.



FIG. 208.—Cayenne, ×110.
Transverse section through seed coat and part of endosperm. Collapsed parenchyma cells separate endosperm from long epidermal cells.

SPICES.



FIG. 209.—Cayenne, ×110.
Surface view of fruit epidermis.

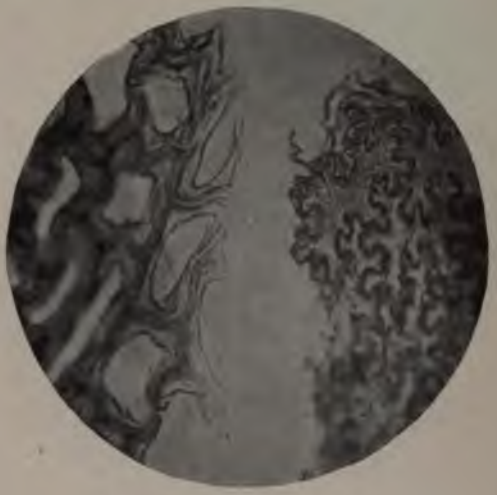


FIG. 210.—Cayenne, ×110.
Surface view of two layers of seed coat.

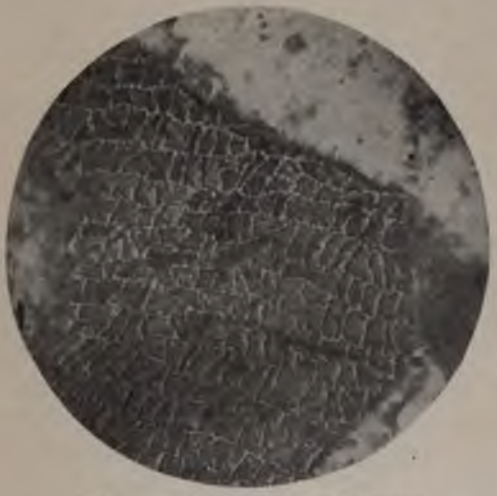


FIG. 211.—Powdered Cayenne, ×110.
A large mass of fruit epidermis.

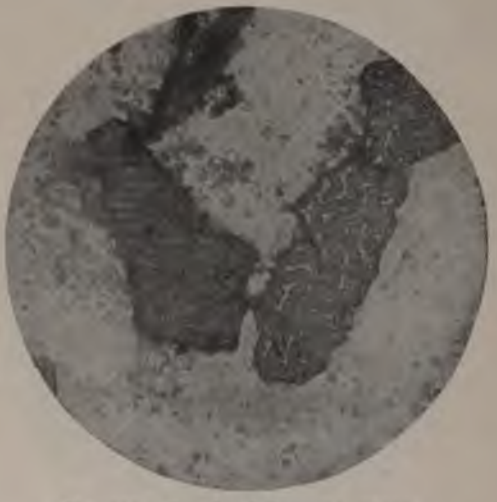


FIG. 212.—Powdered Cayenne, ×110.
Showing chiefly two of the seed coat layers.

SPICES.

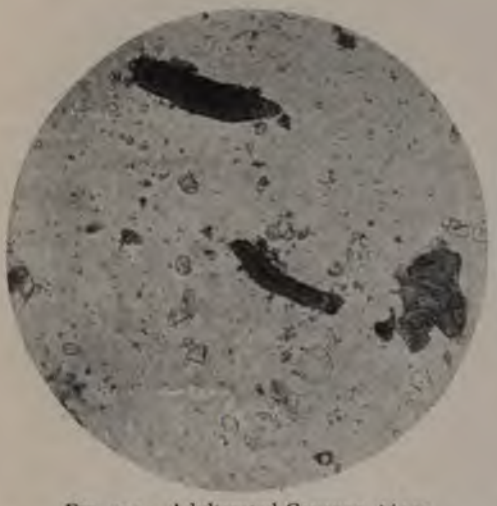


FIG. 213.—Adulterated Cayenne, ×130.
Corn and wheat starch and cocoanut shells appear chiefly. A bit of cayenne is shown at the right.

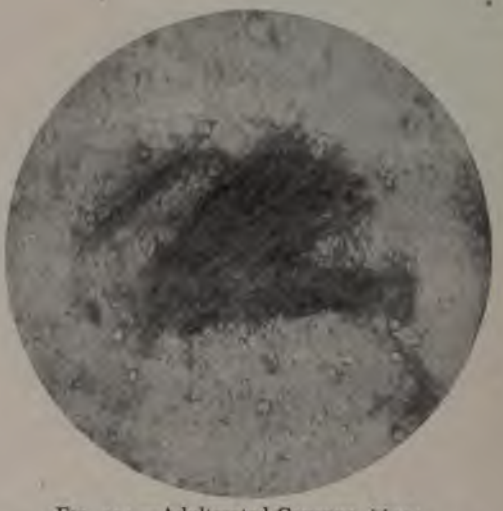


FIG. 214.—Adulterated Cayenne, ×214.
The central mass is ground red wood, surrounded by corn starch grains.

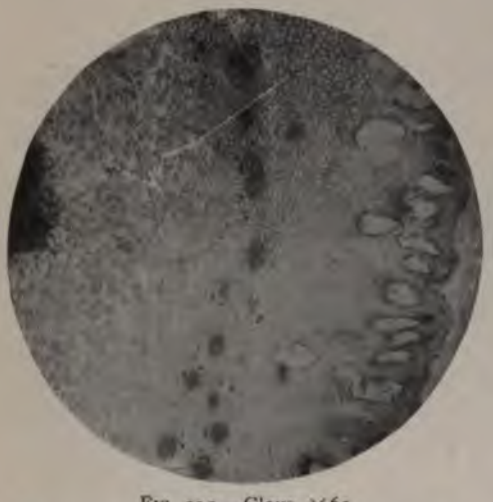


FIG. 215.—Clove, ×65.
Transverse section from the center outward to epidermis, showing parenchyma.



FIG. 216.—Clove, ×110.
Transverse section near epidermis, showing large oil cavities.



SPICES.

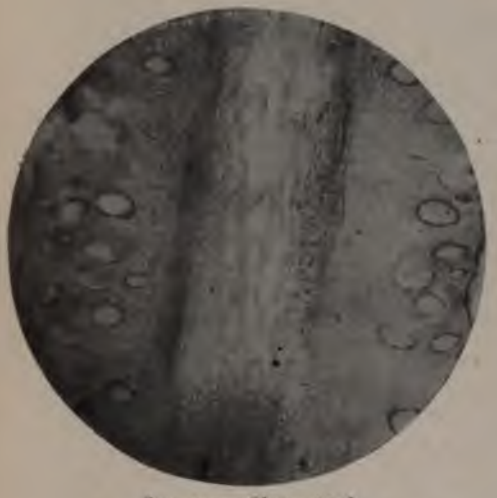


FIG. 217.—Clove, ×28.
Longitudinal section through entire clove.

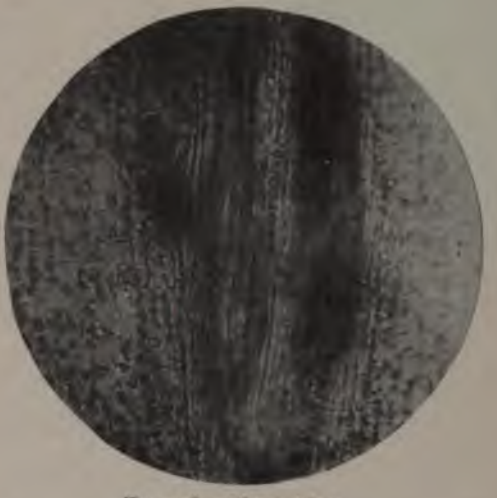


FIG. 218.—Clove, ×70.
Central longitudinal section, showing duct bundles.

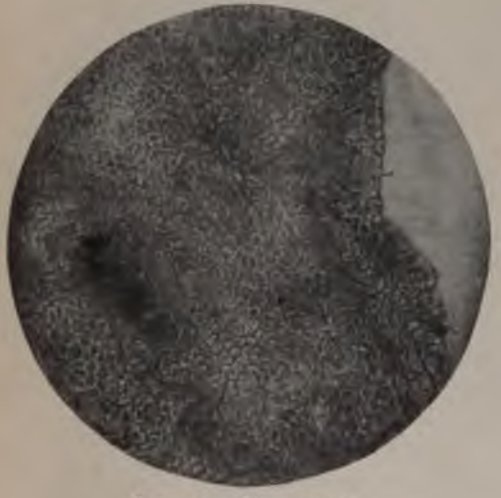


FIG. 219.—Clove, ×110.
Surface view of epidermis

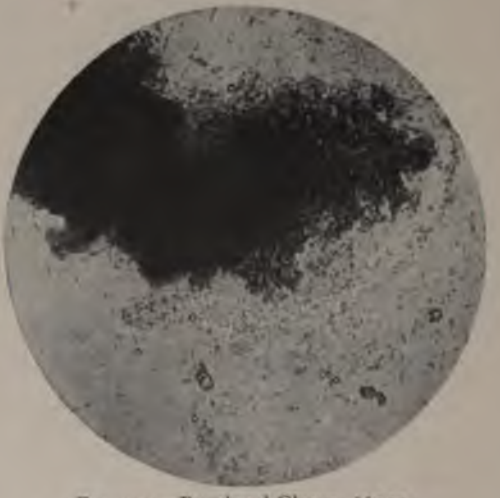


FIG. 220.—Powdered Cloves, ×130.
Dense, spongy tissue, with small oil drops.

SPICES.

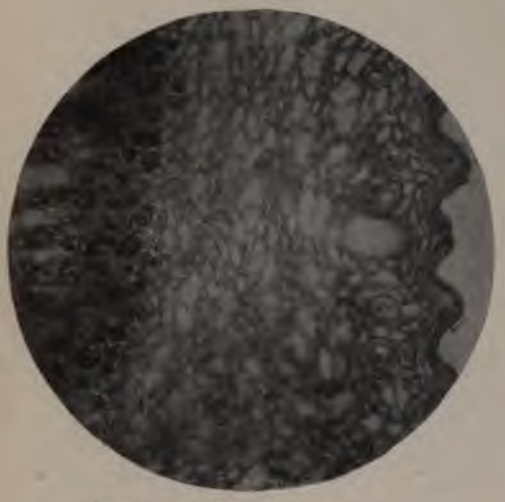


FIG. 221.—Clove Stem, ×70.
Transverse section through outer part of stem, showing bast fibers at the left, parenchyma in the center, and stone cells near the epidermis.



FIG. 222.—Clove Stem, ×25.
Central longitudinal section through entire stem, showing bast fibers in the center, and stone cells at the right.

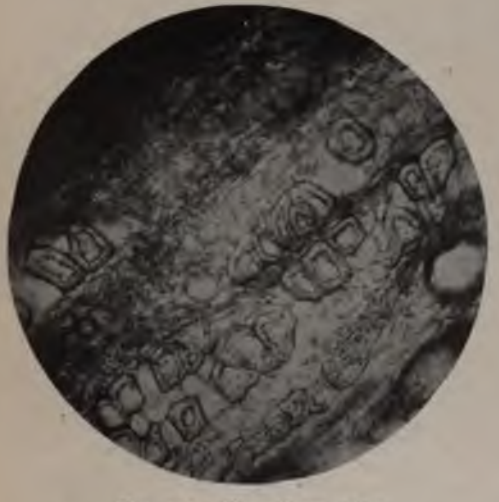


FIG. 223.—Clove Stem, ×70.
Longitudinal section, showing the stone cells.

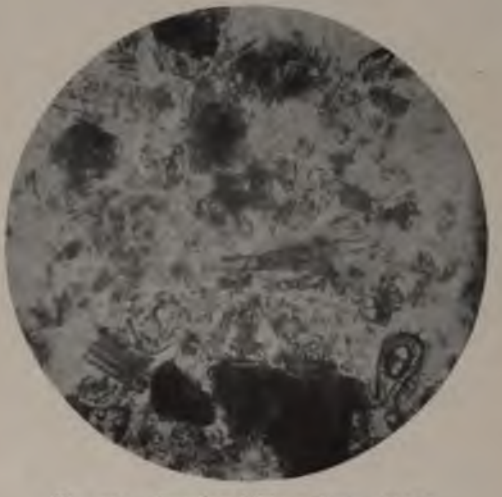


FIG. 224.—Powdered Clove Stems, ×110.
Showing fragments of tissues, stone cells, and bast fibers.

SPICES.

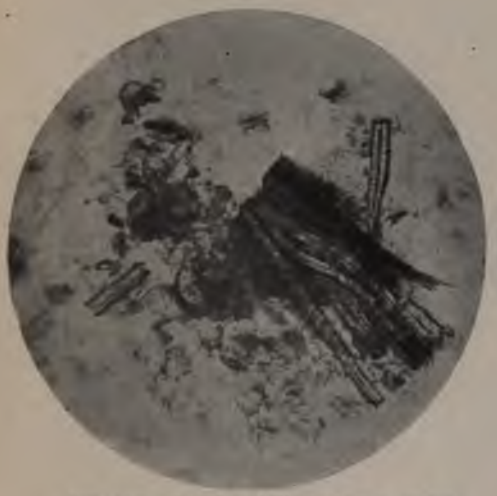


FIG. 225.—Powdered Clove Stems, ×119.
Showing bundle of bast fibers.

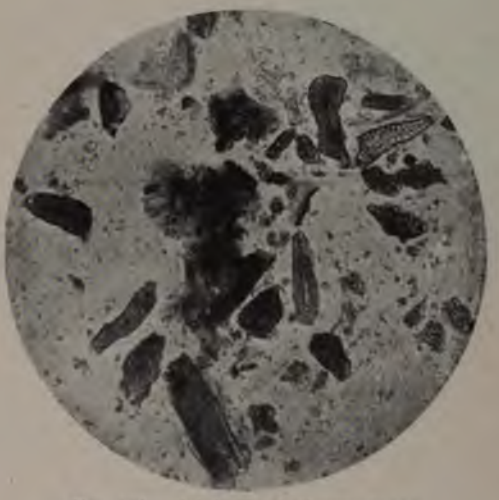


FIG. 226.—Adulterated Cloves, ×130.
Showing chiefly stone cells of cocoanut shells.

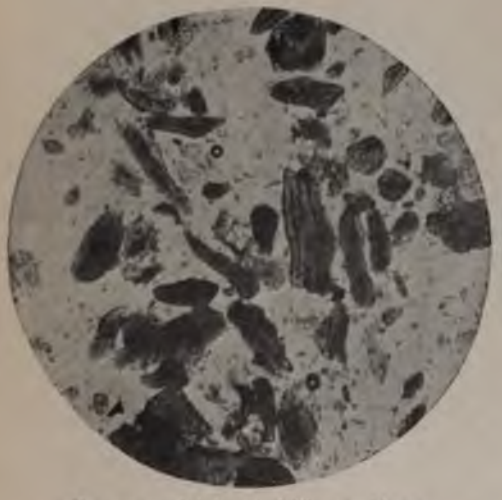


FIG. 227.—Adulterated Cloves, ×130.
With large admixture of cocoanut shells.

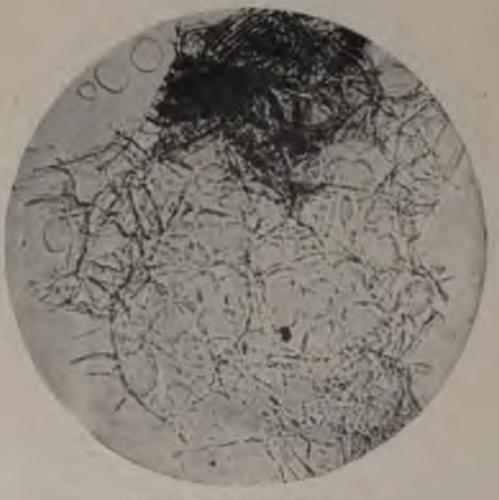


FIG. 228.—Ginger, ×110.
Transverse section, showing starch cells with contents.

SPICES.

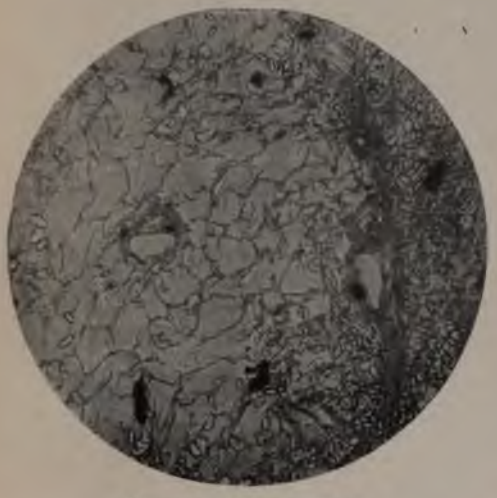


FIG. 229.—Ginger, ×110.
Transverse section, showing parenchyma, starch grains, and duct vessels.



FIG. 230.—Ginger, ×110.
Longitudinal section, showing spiral ducts and pigment cells.

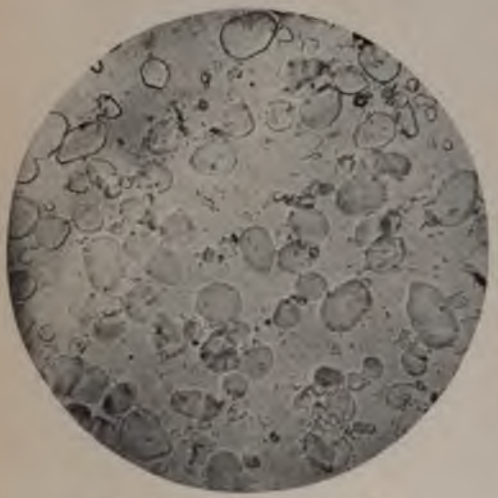


FIG. 231.—Ginger Starch, ×220.

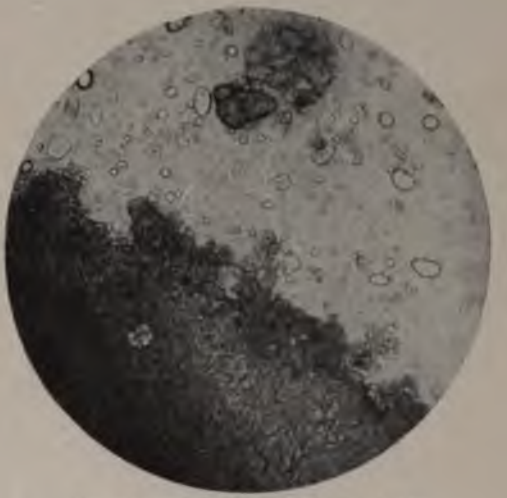


FIG. 232.—Adulterated Ginger, ×130.
A mass of wheat bran tissue is most conspicuous.

SPICES.

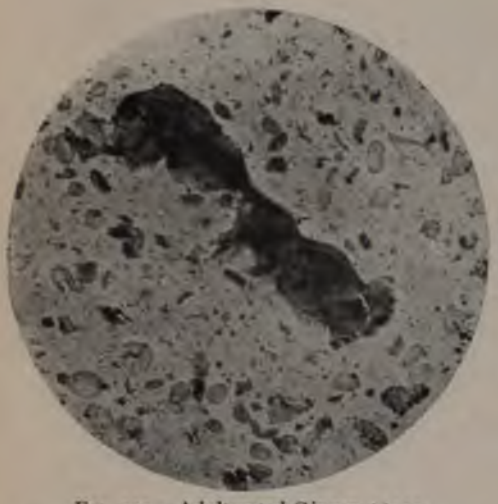


FIG. 233.—Adulterated Ginger, ×130.
The central dark mass is a yellow fragment of turmeric.

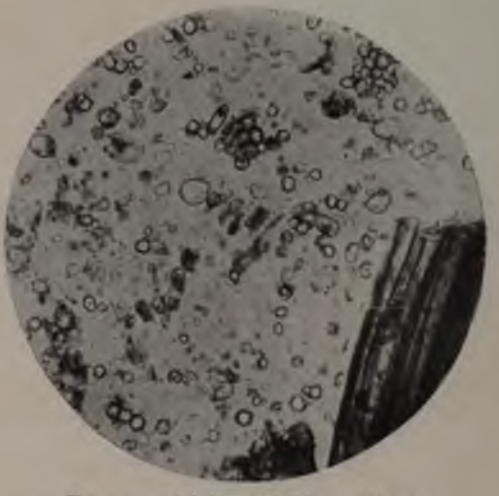


FIG. 234.—Adulterated Ginger, ×130.
Containing a large admixture of corn and wheat starches



FIG. 235.—Penang Mace, ×110.
Transverse section through epidermis and oil cells, showing also parenchyma with contents of amylodextrin.



FIG. 236.—Bombay or Wild Mace, ×110.
Transverse section through outer layers, showing yellow and red resinous lumps.

SPICES.

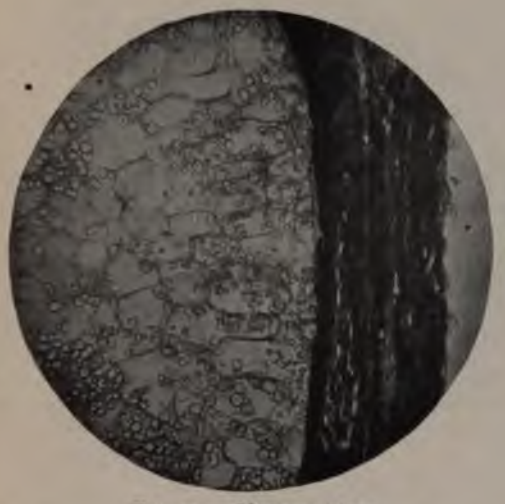


FIG. 237.—Nutmeg, ×110.
Transverse section through the exterior and interior teguments of the seed and part of the endosperm, showing starch cells.

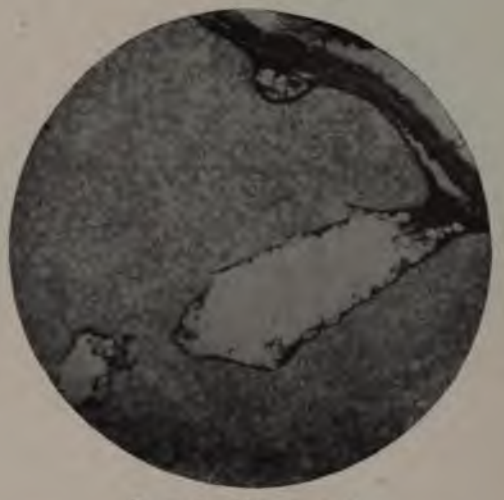


FIG. 238.—Nutmeg, ×25.
Transverse section near exterior of seed.

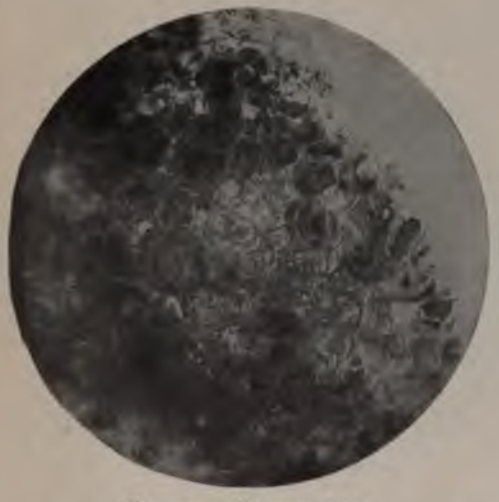


FIG 239.—Nutmeg, ×110.
Surface view of seed coat, showing also portions of underlying tissues.

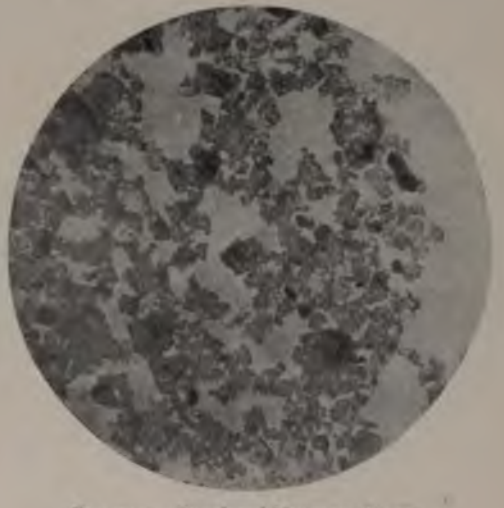


FIG. 240.—Powdered Nutmeg, ×110.

SPICES.

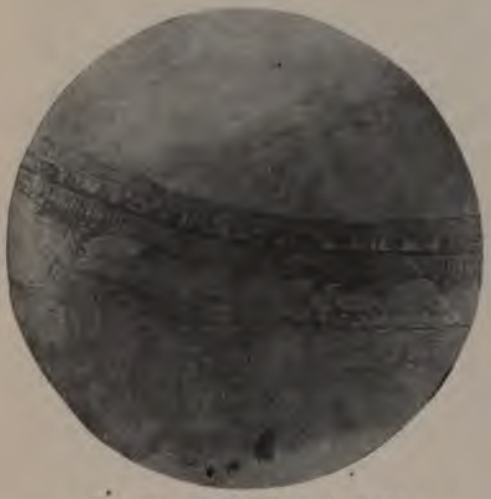


FIG. 241.—White Mustard, ×110
Transverse section through mucilaginous epidermis, sub-epidermal parenchyma layer (square cells), palisade cells, and broken parenchyma layer of the hull.



FIG. 242.—White Mustard, ×110.
Transverse section through the tissue of the radicle.

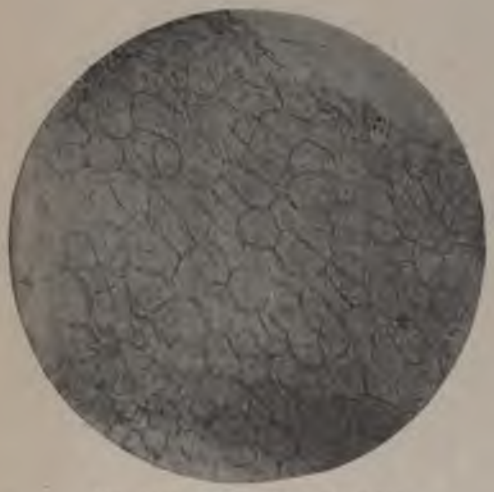


FIG. 243.—White Mustard ×110
Surface view of two layers of the hull or seed coat.



FIG. 244.—White Mustard. ×110.
Surface section through palisade cells and underlying layer of the seed coat.

SPICES.

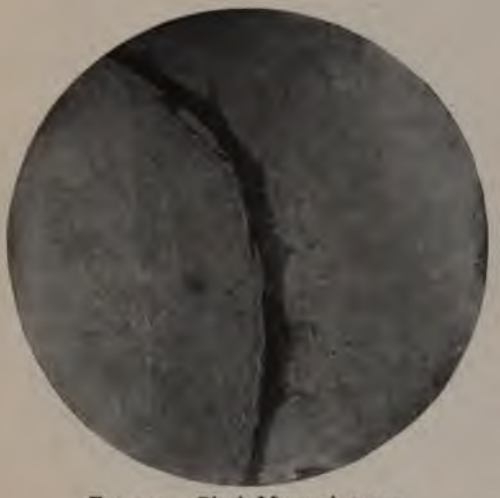


FIG. 245.—Black Mustard, ×110.
Transverse section, showing fragments of the epidermis and dark colored palisade cells of the seed coat.

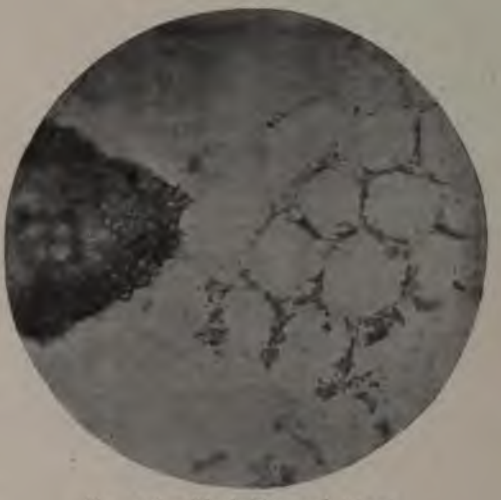


FIG. 246.—Black Mustard, ×110.
Surface view of two of the seed coat layers.

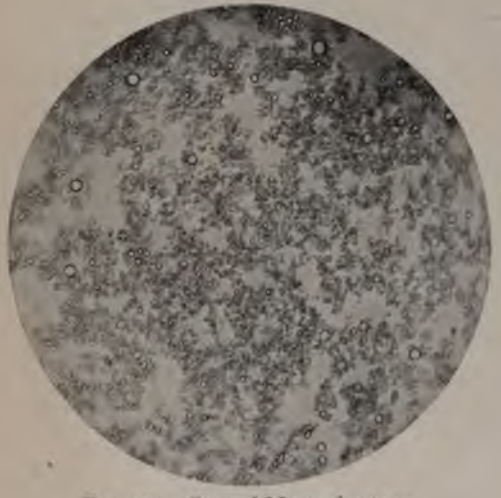


FIG. 247.—Ground Mustard, ×130.
Ground without removal of the oil.

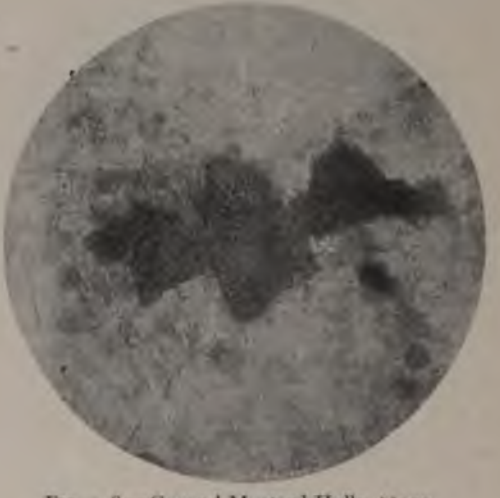


FIG. 248.—Ground Mustard Hulls, ×110

SPICES.



FIG. 249.—Dakota Mustard Flour, ×110.
Dark spots show starch grains of foreign weed seed, stained with iodine.

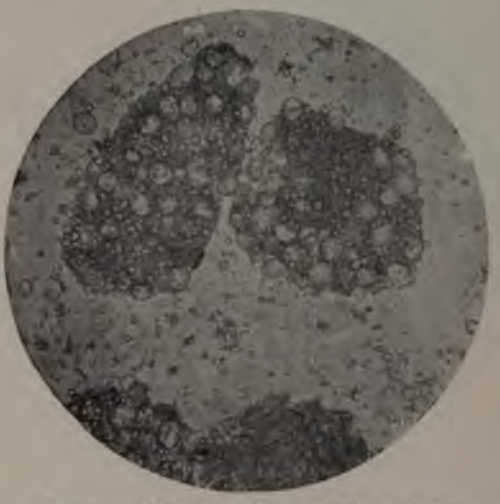


FIG. 250.—Adulterated Mustard Flour, ×130.
Showing masses of wheat starch.

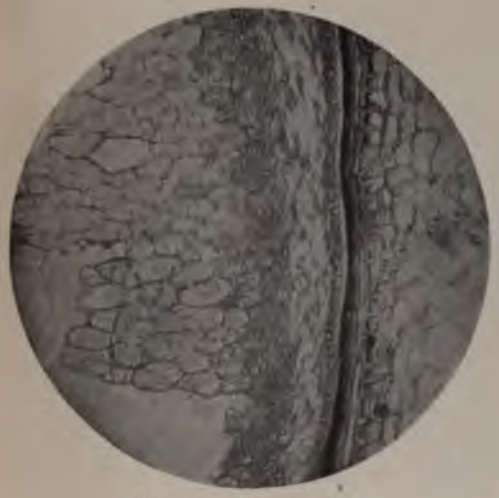


FIG. 251.—Pepper, ×110.
Transverse section through inner part of pericarp (including parenchyma and seed coat layers) and portion of perisperm, showing starch and oil cells.



FIG. 252.—Pepper, ×110.
Surface view of hypodermal layer.

SPICES.



FIG. 253.—Pepper, ×110.
Transverse section through outer part of pericarp, showing epidermis, underlying stone cell layers, parenchyma, and seed coat.

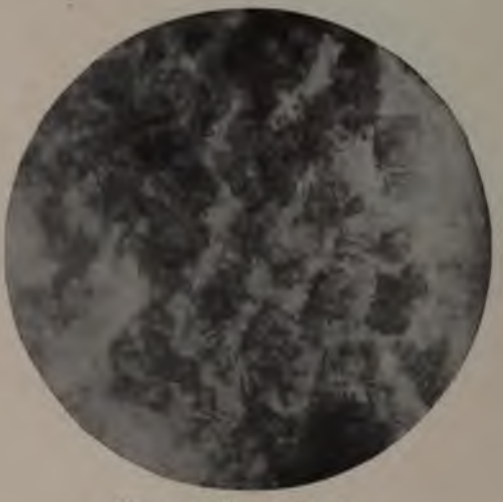


FIG. 254.—Pepper, ×110.
Surface section through stone cell layer.

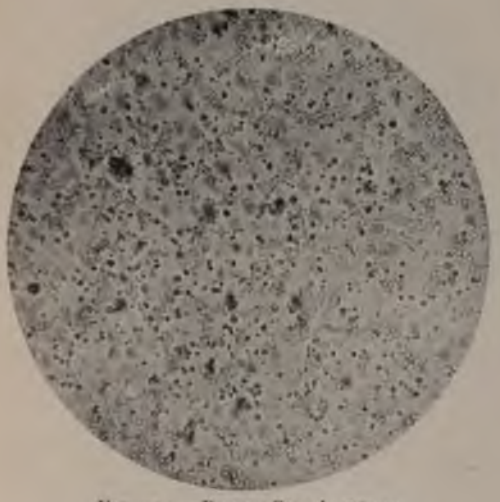


FIG. 255.—Pepper Starch, ×220.
Starch granules separated.

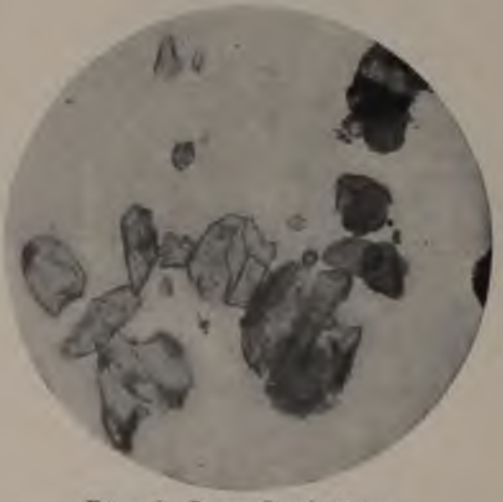


FIG. 256.—Pepper Starch, ×110.
Starch grains in masses.

SPICES.

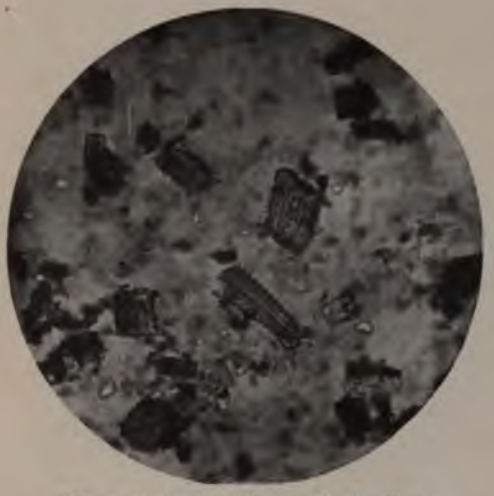


FIG. 257.—Ground Pepper Shells, ×110.
Mainly showing stone cells.



FIG. 258.—Adulterated Pepper, ×130.
Showing wheat and buckwheat starches.

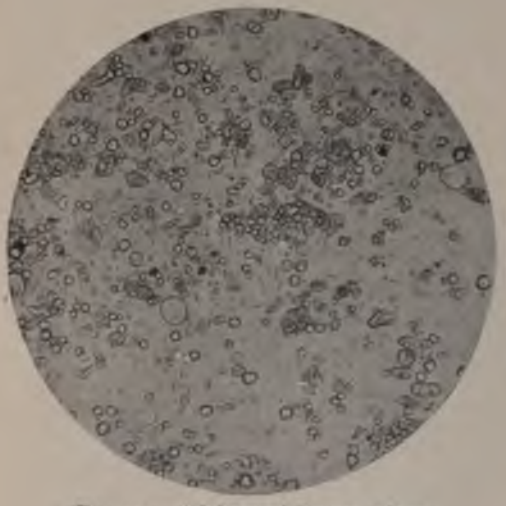


FIG. 259.—Adulterated Pepper, ×130.
Showing wheat, corn, and rice starches.

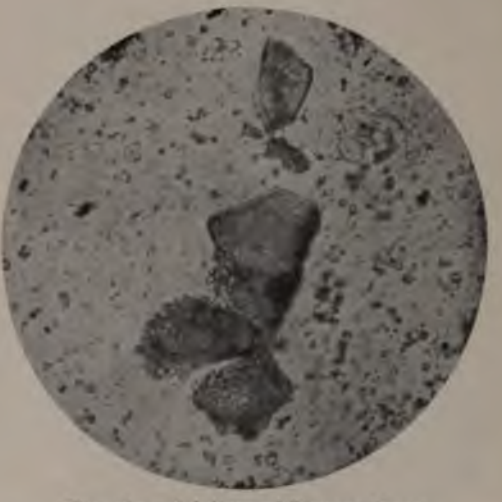


FIG. 260.—Adulterated Pepper, ×130.
The large, lower mass shows buckwheat starch, while the finer-grained mass near the top is of pepper.



SPICES. SPICE ADULTERANTS.

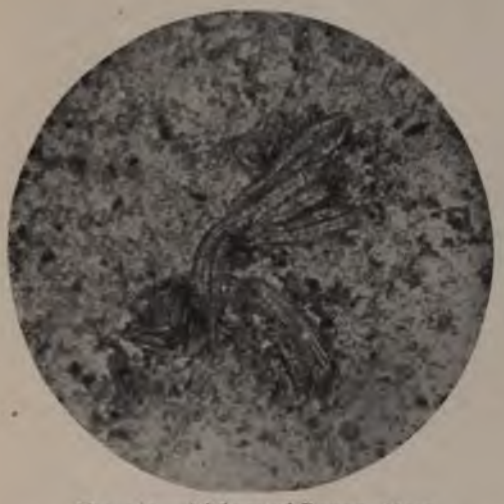


FIG. 261.—Adulterated Pepper, ×110.
The central mass shows the sclerenchyma cells of olive stones.

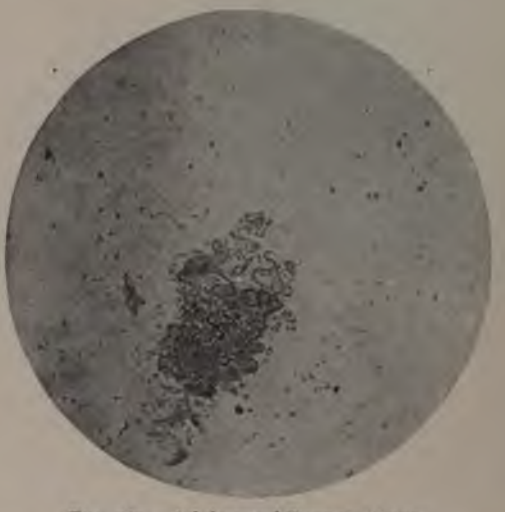


FIG. 262.—Adulterated Pepper, ×130.
Cayenne and wheat starch are the adulterants.

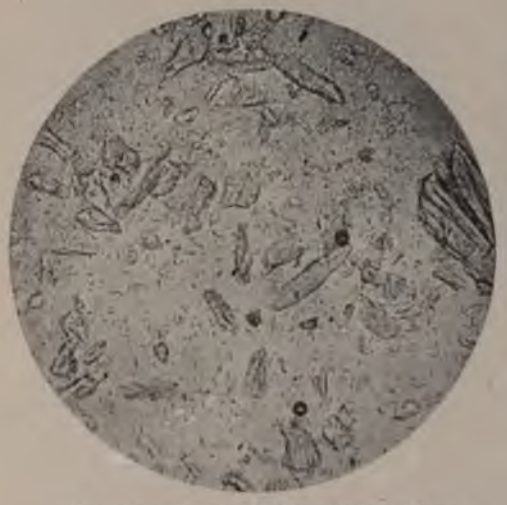


FIG. 263.—Powdered Olive Stones, ×110.

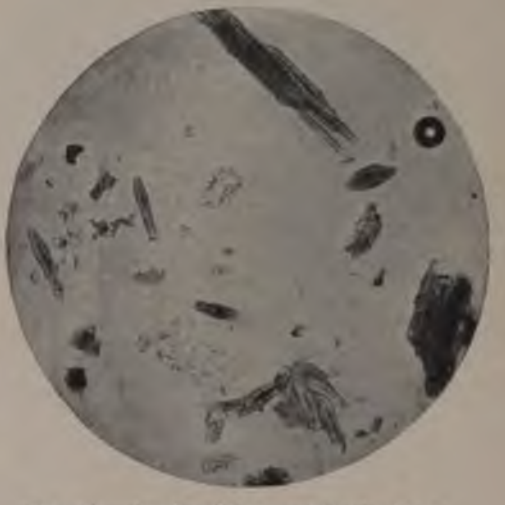


FIG. 264.—Powdered Cocoanut Shells, ×110

SPICE ADULTERANTS.

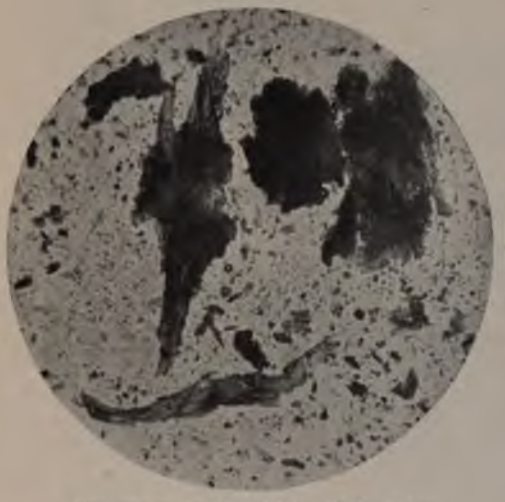


FIG. 265.—Powdered Elm Bark, ×110.

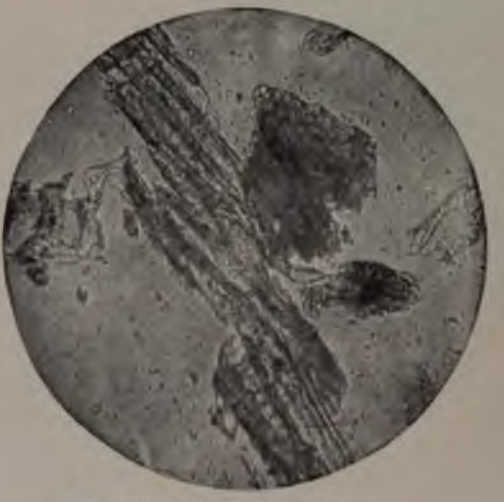


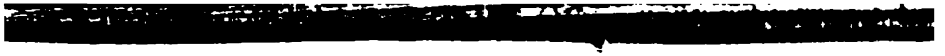
FIG. 266.—Pine Sawdust, ×110.
Finely ground.



FIG. 267.—Pine Wood, ×110.
Transverse section.



FIG. 268.—Pine Wood, ×110
Radial and tangential sections.



EDIBLE FATS.



FIG. 269.—Pure Butter, ×25.
With polarized light and selenite plate.



FIG. 270.—Process or Renovated Butter, ×25.
With polarized light and selenite plate.

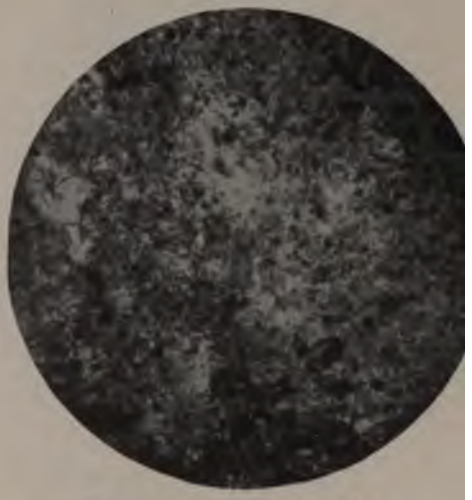


FIG. 271.—Oleomargarine, ×25.
With polarized light and selenite plate.

EDIBLE FATS.



FIG. 272.—Lard Stearin, ×110.
Leaf lard, crystallized from ether.

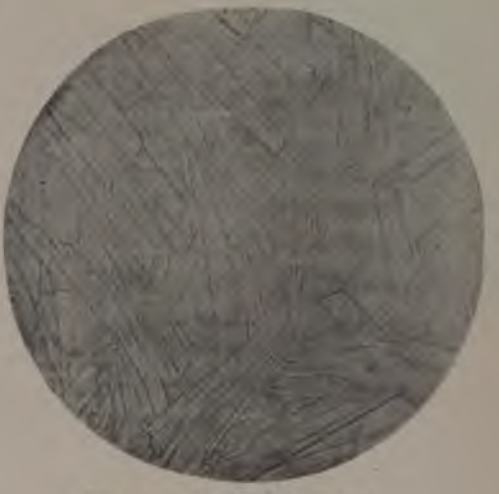


FIG. 273.—Lard Stearin, ×220.
Leaf lard, crystallized from ether.



FIG. 274.—Lard Stearin, ×220.
"Back" lard, crystallized from ether.

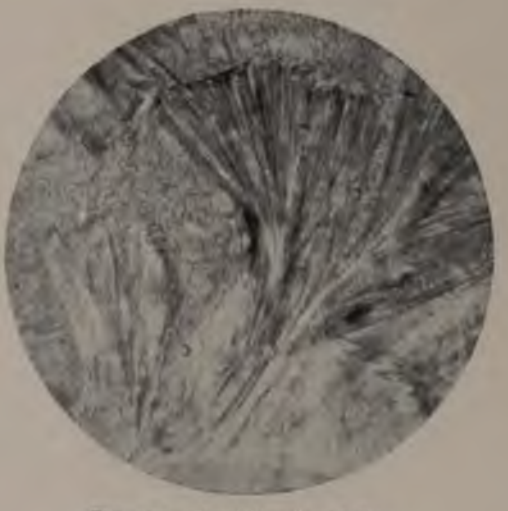


FIG. 275.—Lard Stearin, ×480.
"Back" lard, crystallized from ether

EDIBLE FATS.

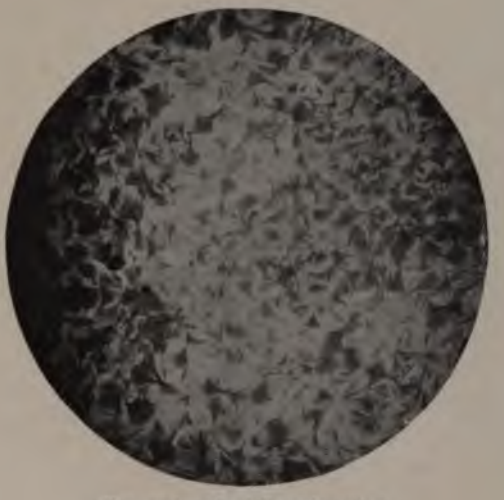


FIG. 276.—Beef Stearin, ×35.
Crystallized from ether.

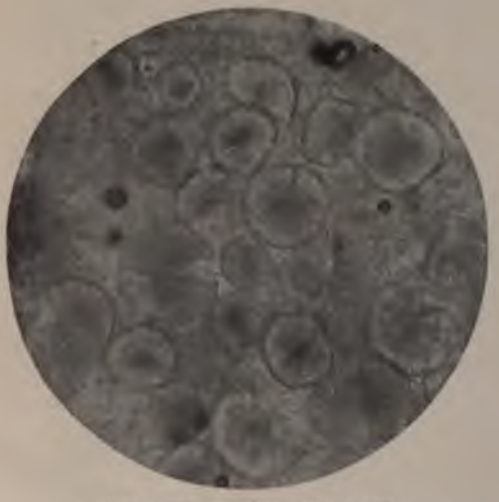


FIG. 277.—Beef Stearin, ×110.
Crystallized from ether.

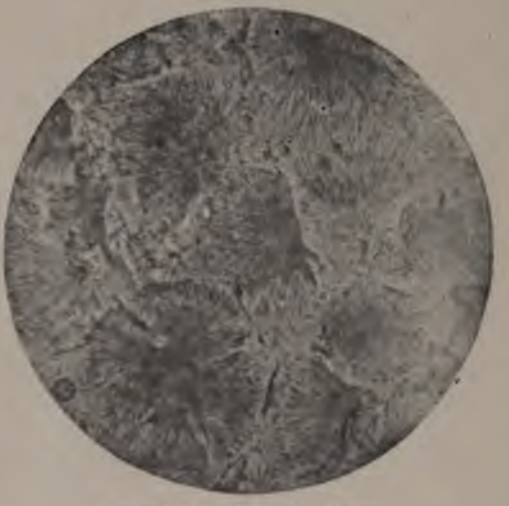


FIG. 278.—Beef Stearin, ×220.
Crystallized from ether.



